THE EXTRACTION OF BENZOIC ACID IN A PULSE COLUMN

by

CHANG-LO WU

B.S., The Ordnance Engineering College
Taipei, Formosa, 1953

A THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Chemical Engineering

KANSAS STATE COLLEGE
OF AGRICULTURE AND APPLIED SCIENCE

1958

INTRODUCTION

In order to have highly efficient methods of separating materials by solvent extraction, different types of columns have been developed. The purpose of this research is to study the characteristics of a pulse column and relationship between H.T.U. and other variables such as amplitude, frequency and flow rate. With such a column it is possible to separate copper from cadmium or uranyl nitrate from dilute solutions for purification. This study was conducted to obtain a method of separation for high purification and efficiency. Theoretically, this type of pulse operation has some principal advantages, due to its high degree of dispersion and turbulence, which usually leads to a reduction in column height. Also, lower stage heights are obtained when compared with conventional packed towers. It is, therefore, of great advantage when the process contains radioactive materials, since shielding costs will be reduced because the pulse characteristic can be varied externally to the column and operational flexibility will be obtained.

The extraction of benzoic acid by toluene used in this research is a simple process by which the characteristics of a pulse column may be determined. A simple analytical method to determine quantitatively the benzoic acid in toluene and water was followed.

In a column of this type the pulse is normally produced by use of displacement or diaphragm pumps. A special method of pulsing was devised for this column with the installation of air operated bellows, both on the top and bottom of the column so that frequency, amplitude and wave form could be varied rapidly at will. The control arrangement consisted of an electronic oscillator which actuated the solenoid valves.

Research work using pulse columns to extract material started early in 1953. A patent issued to Van Dijck (18) described a process which used a pulsing action to force liquid through holes in a sieve plate alternately in opposite directions. Information on another column of this type is contained in a 1949 report by Burns, et al. (3) who coined the name pulse column. At the same time Van Berg and Wiegandt (17) began a program to study the use of externally applied energy as an aid in separation process. In 1950 declassified reports pertaining to this subject and published by the Atomic Energy Commission appeared in the public literature. Griffith, et al. (7) are the authors.

More recently liquid-liquid extraction on the same subject was reported in March of 1957 by Li and Newton (10) at Georgia Institute of Technology in which pulsing in the column was accomplished by the proportioning feed pumps. This type of operation produced discontinuous injection of solvent and feed material and therefore differed from this program. Other differences were the nature of column construction and materials and the type of liquid-liquid contact which was effected.

EQUILIBRIUM STUDIES

Equilibrium distribution studies of benzoic acid in water and toluene at various temperatures have been made. The chemical procedure for equilibrium determination was as follows: Several different solutions of low concentration benzoic acid were prepared in 100 cc volumetric flasks. A small amount of heating was required to effect complete solution. The concentrations of the solutions prepared were 0.1 N, 0.08 N, 0.05 N, 0.02 N, 0.01 N and 0.005 N. Twenty-five cc samples of each concentration were pipeted into separatory funnels. To each funnel was added 25 cc of pure

toluene, followed by vigorous shaking. Observations were made on various temperatures by using a controlled temperature water bath. When equilibrium was reached after the layers had separated, the water phase was withdrawn, and its volume was measured. Similarly the volume of the toluene phase was measured. Titration of both phases (toluene phase and water) with standard sodium hydroxide using phenolphthalein as indicator was reported. Data are shown in Table 1 and plotted on Plate I.

EQUIPMENT AND CONSTRUCTION OF COLUMN

The pulse extraction column used in this research study was made up of two two-foot sections of a two-inch inside diameter glass tubes. Within the column was installed a series of perforated plates which extended approximately three feet. At each end of the two-inch glass column a four-inch diameter T-shaped glass section was attached. Teflon bellows in the horizontal T-sections were used to produce the pulsations (Plates II, III, IV). These bellows were pushed back and forth by compressed air through suitable connections to solenoid valves shown in Plate V. The amplitude of the bellows was changed by adjusting the pressure in the air line. The pulse frequency was controlled by a cycle switch on the timer (Plate VI). In Plate VII a portion of the perforated plate section is shown. Total length of the column was 6.5 feet with total hold up 9.24 liters. The two two-foot sections were connected by metal rings with tygon tubing as gaskets. The perforated plates were made of stainless steel with 220 one-thirty-second-inch holes in each plate. Each plate was fixed between two stainless steel tubes with two-inch spacings. The perforated plates and tubes were aligned by a stainless steel rod which was fixed in the cover plates for final alignment. The percent of free area for each plate is 5.4 percent.

Table 1. Equilibrium distribution data (benzoic acid-toluene-water system)

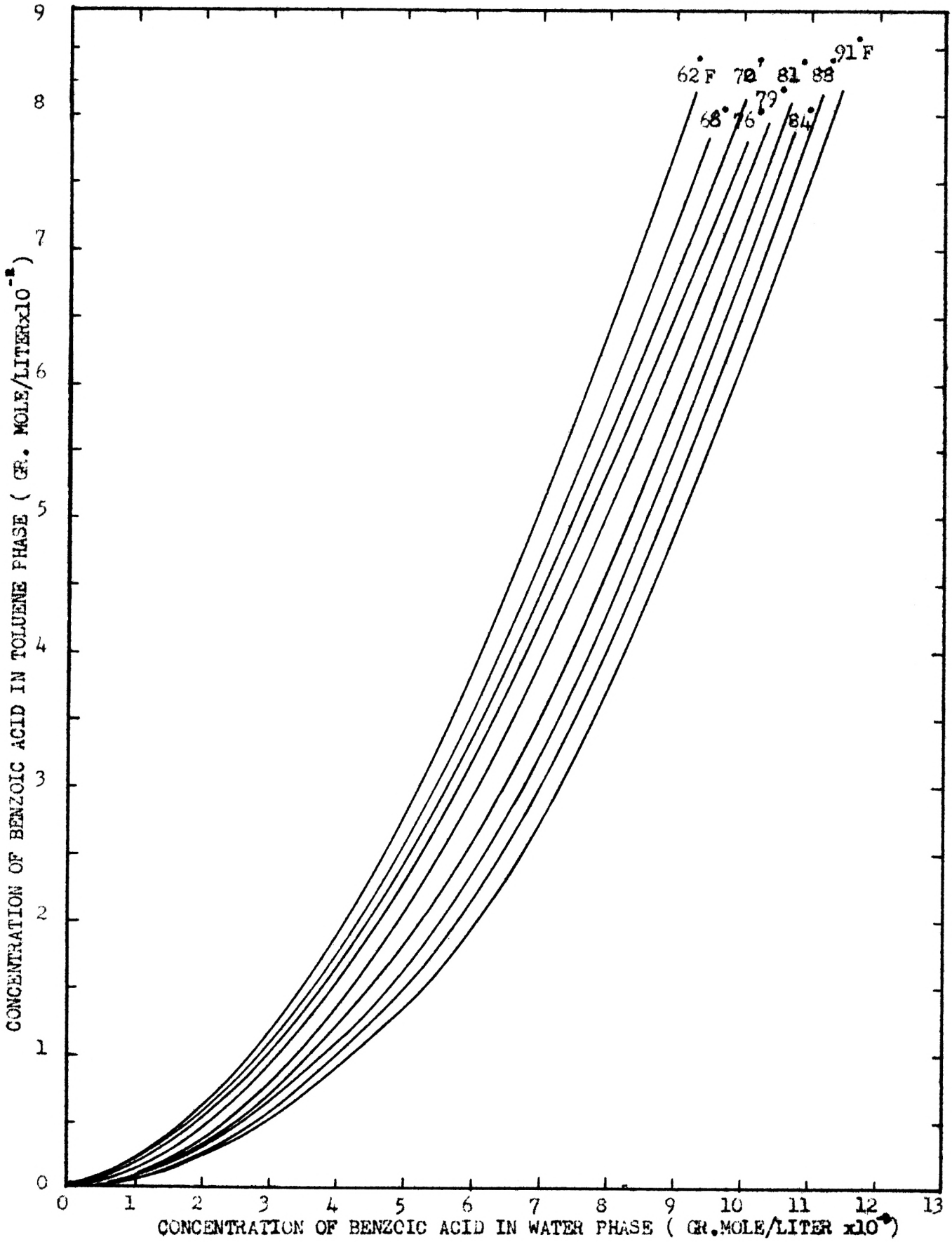
Ct*	Cw**	Ct	Cw
62°F		79°F	
0.0418740	0.0060319	0.236	0.017365
0.0148553	0.003639	0.015475	0.014137
0.0096709	0.002490	0.01432	0.01348
0.0063808	0.001695	0.1288	0.01304
0.0007976	0.0006979	0.1133	0.012375
0.00004985	0.0002991	0.08618	0.01060
		0.071846	0.00994
		0.05799	0.00905
68°F		0.02883	0.006632
0.040877	0.0065802	0.01326	0.004646
0.148850	0.0037886		
0.007278	0.0024925	84°F	
0.002692	0.0016942	0.2321	0.01760
0.000698	0.00064805	0.1898	0.01652
		0.14898	0.01415
		0.09387	0.01150
72°F		0.07673	0.01050
0.41535	0.0062811	0.04774	0.00846
0.015254	0.0037886	0.02851	0.00668
0.007727	0.0025922	0.02587	0.00663
0.004237	0.0021934	0.01450	0.00511
0.0001545	0.0003988	0.01240	0.00456
76°F		91°F	
0.057128	0.0085724	0.2435	0.01906
0.0405779	0.06680	0.1618	0.01570
0.0157526	0.004038	0.1330	0.01435
0.006979	0.002642	0.1166	0.01326
0.0032402	0.0018943	0.1027	0.01281
0.000648	0.0007976	0.0873	0.01171
		0.05911	0.00972
		0.05863	0.00972
		0.02867	0.00708
		0.01437	0.005094

* Ct = Concentration of benzoic acid in toluene phase (moles/liter)
** Cw = Concentration of benzoic acid in water phase (moles/liter)

EXPLANATION OF PLATE I

Equilibrium Distribution of Toluene, Benzoic, Water System at Room Temperature for Dilute Solution

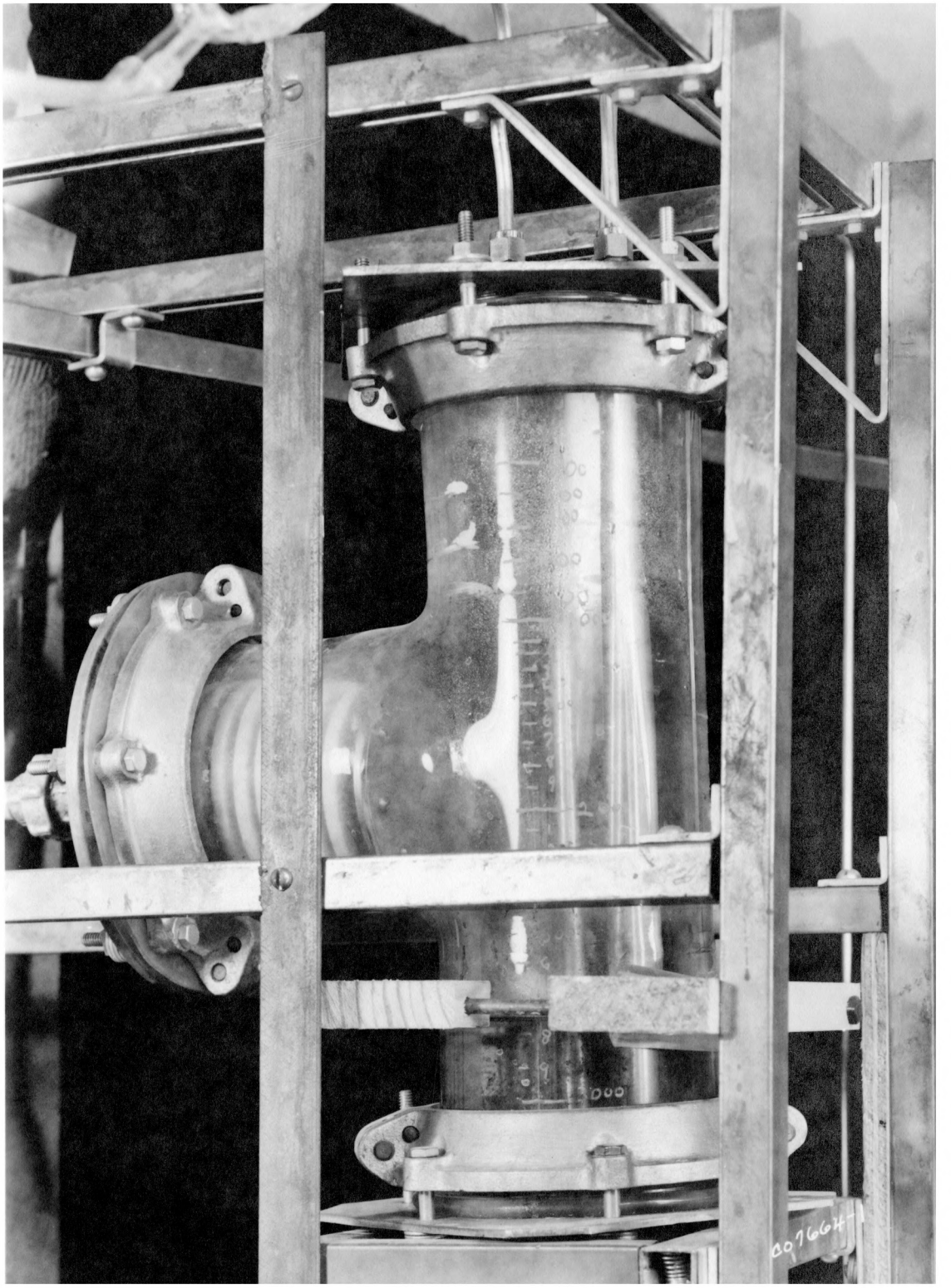
PLATE I


62°F
72°
81°
88°
91°F
68°
76°
79°
84°
CONCENTRATION OF BENZOIC ACID IN TOLUENE PHASE (GR. MOLE/LITER x10⁻²)
CONCENTRATION OF BENZOIC ACID IN WATER PHASE (GR.MOLE/LITER x10⁻⁴)
0
1
2
3
4
5
6
7
8
9
10
11
12
13

EXPLANATION OF PLATE II

The view of connection between teflon bellows and the horizontal T section at the top of the column

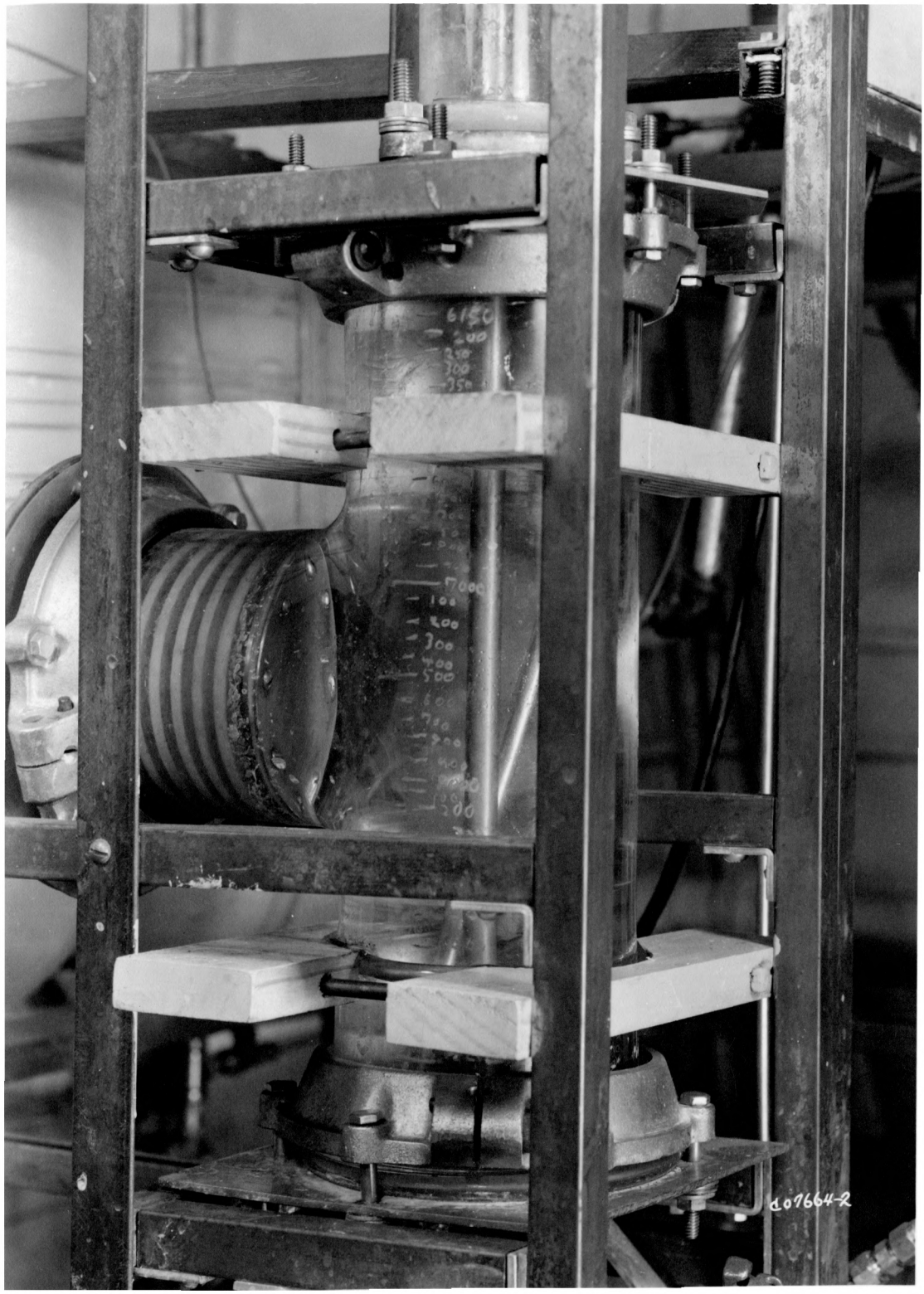
PLATE II



EXPLANATION OF PLATE III

The view of connection between the teflon bellows and the horizontal T section at the bottom of the column

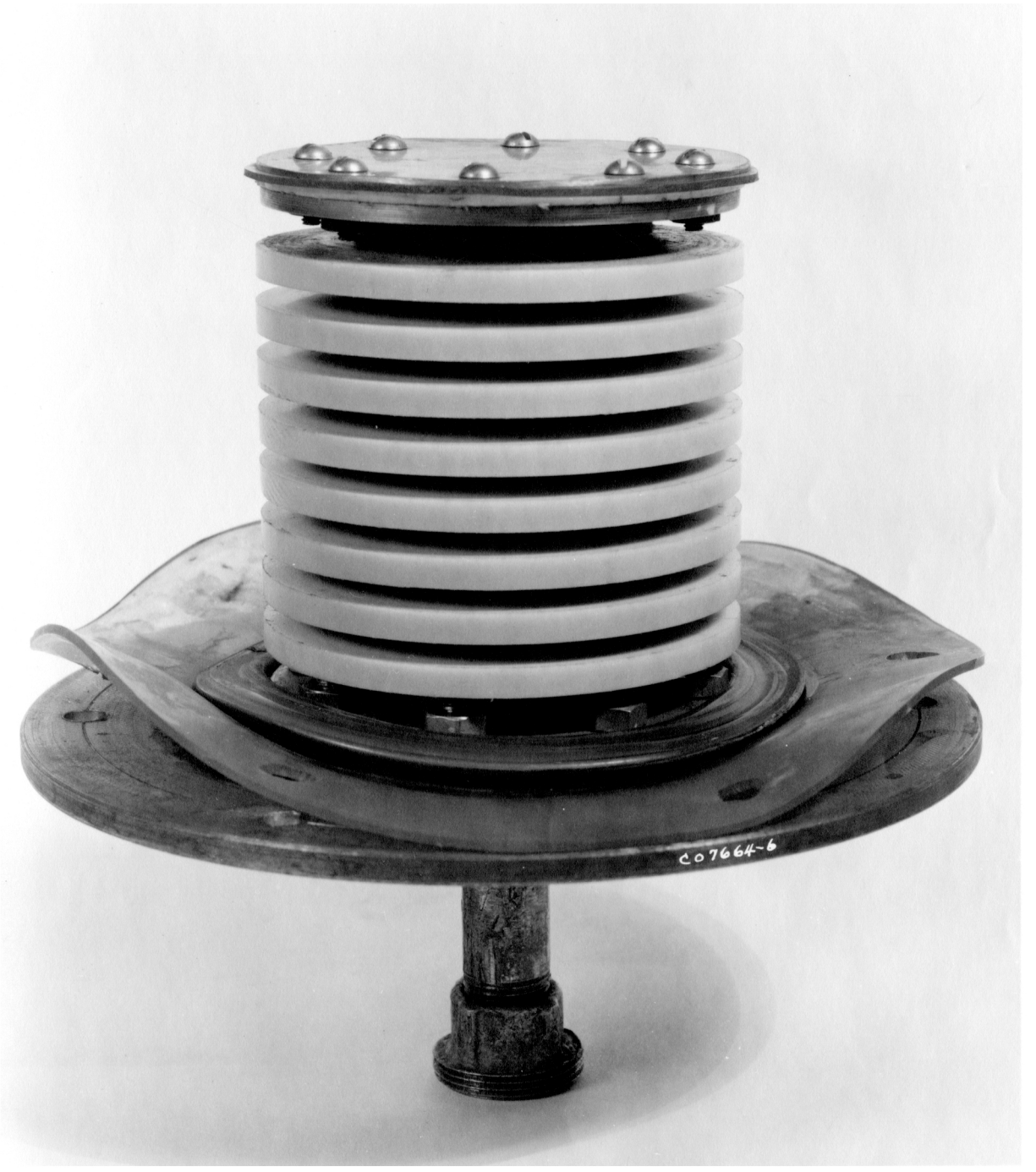
PLATE III



EXPLANATION OF PLATE IV

A view of the teflon bellows

PLATE LV

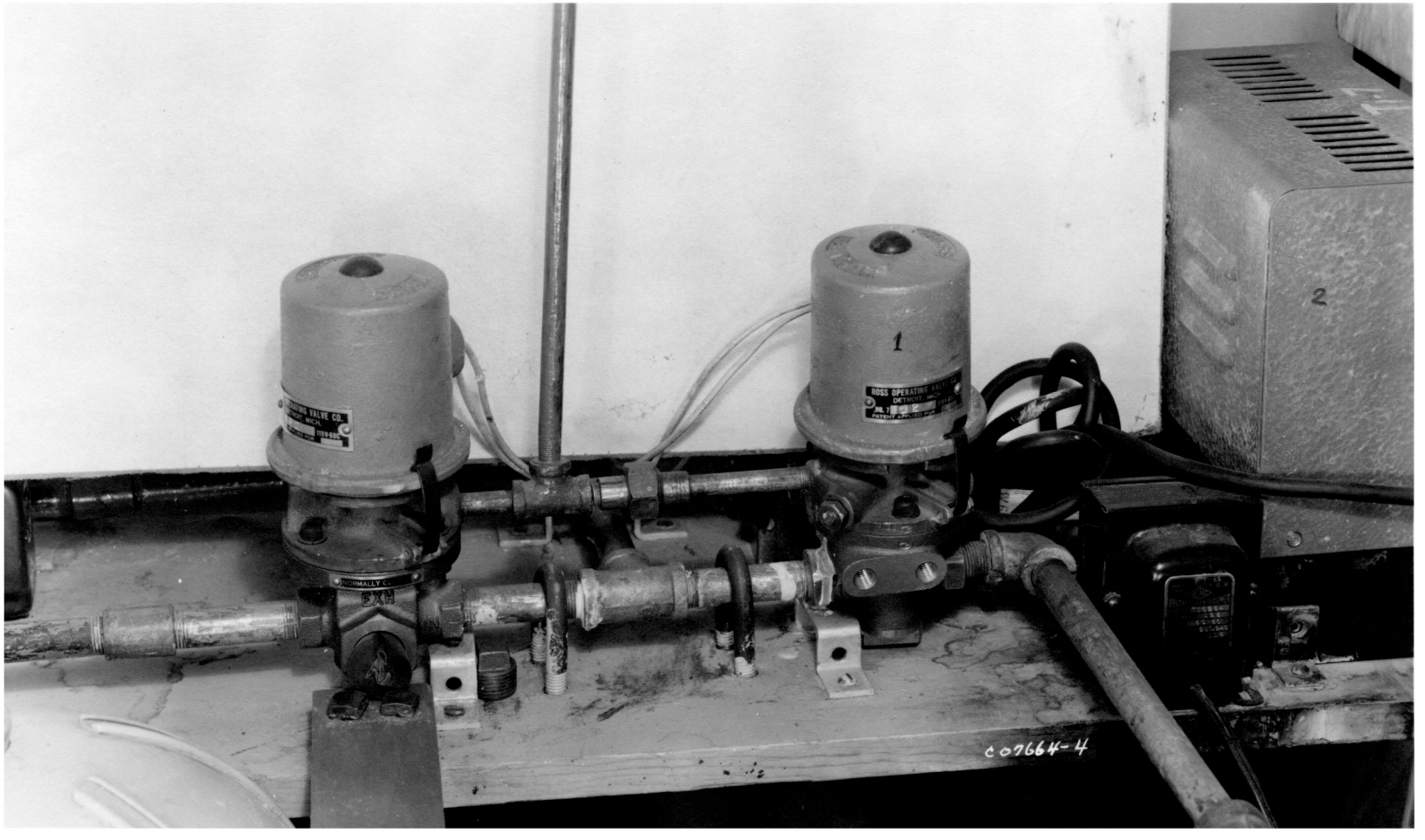


EXPLANATION OF PLATE V

A view of solenoid valves

1. The solenoid valves
2. Timer

PLATE V

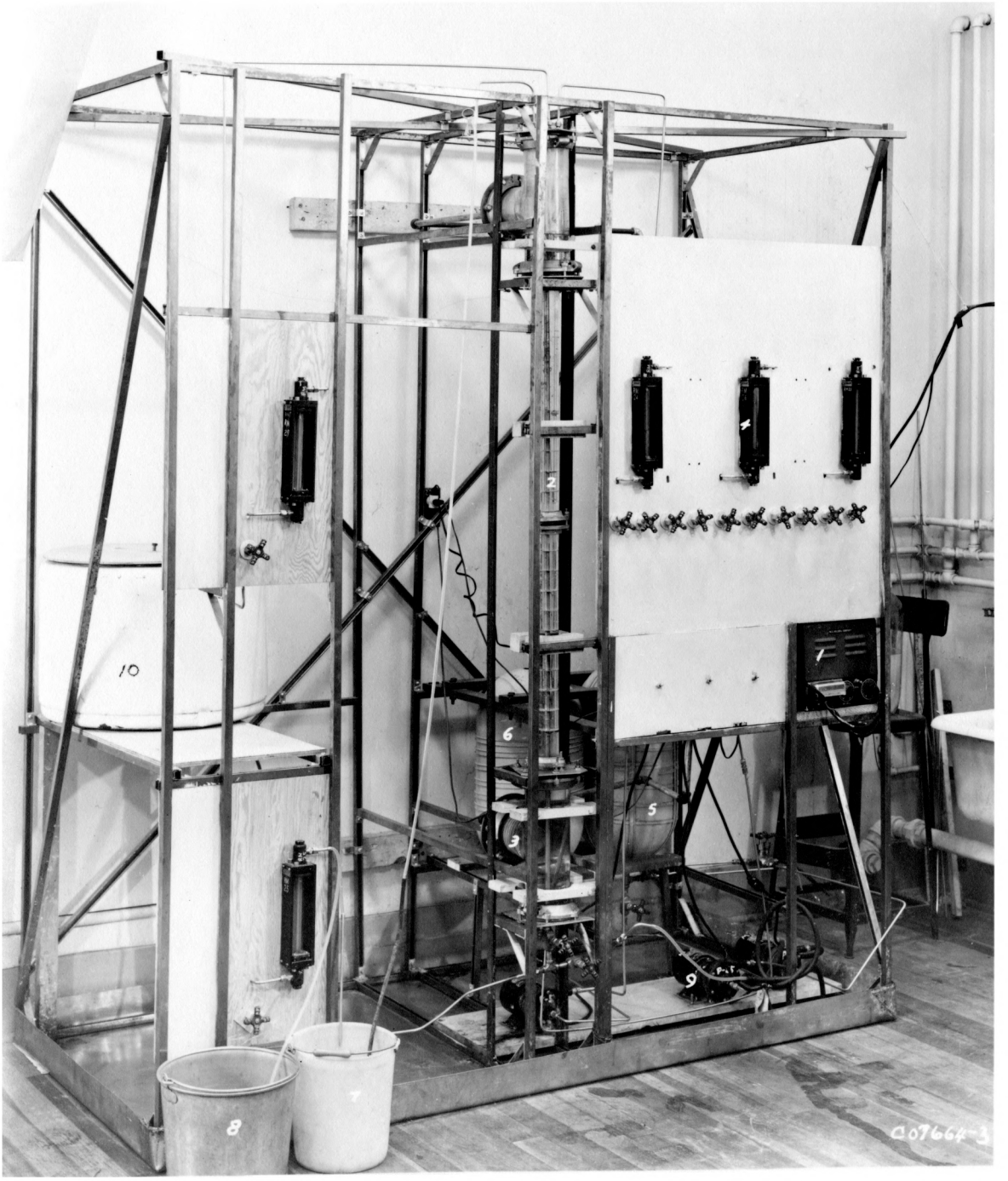


EXPLANATION OF PLATE VI

View of the Whole Pulse Column

1. Timer
2. The Pulse Column
3. Teflon Bellows
4. Flowrator for Light Solvent
5. Pure Toluene Tank
6. Feed Solution Tank
7. Toluene Receiver
8. Water Receiver
9. Light Solvent Centrifugal Pump
10. Feed Solution Storage Tank
11. Flowrator for Feed Solution

PLATE VI

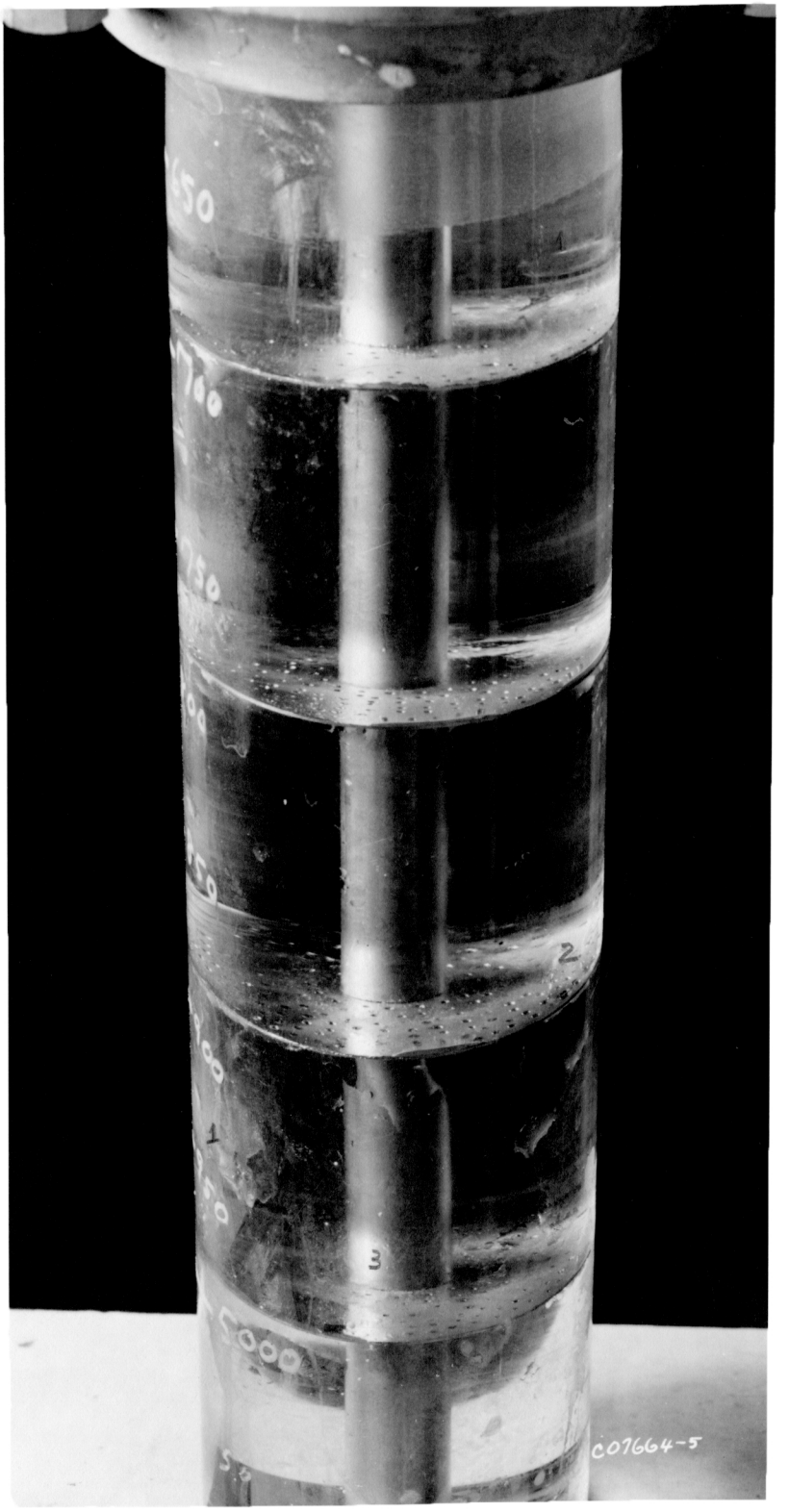


EXPLANATION OF PLATE VII

A closer view of two-inch column

1. Two-inch glass column
2. Perforated plate
3. Stainless steel tube

PLATE VII



Two 40-liter stainless steel tanks were used for storing the feed solution and solvent (Plate VI). The feed units consisted of two one-half horse power centrifugal pumps for maintaining a continuous flow and two Fischer-Porter flowrators for indicating the rate of the liquid feed. Each of the two streams were delivered to the column at points which were located about 13 inches above and below the bottom and top coverplates. Valves and flowrators were suitably connected with the extract and raffinate flow lines.

A diagram of the system is shown in Plate VIII. All of the connecting lines were ¼-inch stainless steel tubing. The ¼-inch control needle valves and threaded tube adaptor fittings were of 344 stainless steel. The connections between the top outlet of the column and its product receiver was ¼-inch tygon tubing. A one hundred liter tank was employed as aqueous feed storage reservoir and two five-gallon stainless steel tanks were used as product collectors. The frequency control units consisted of a timer, together with a relay which was used to reduce its load. It is indicated on Plates V and VI. The compressed air was dried before it reached the solenoid valves and bellows.

METHOD OF OPERATION

Run Procedure

Two things were of most importance in regard to operating the column: one, start-up, and the other, control of the interface. For start-up, the water solution was pumped into the bottom until one-third of the column was filled. Continuous operation was established by feeding water solution at the top and toluene at the bottom. The water solution in the column at start-up was removed slowly in order to approach equilibrium condition within the pulse column. Usually equilibrium conditions were reached when two

EXPLANATION OF PLATE VIII

Flow Sheet of Continuous Extraction of Benzoic Acid

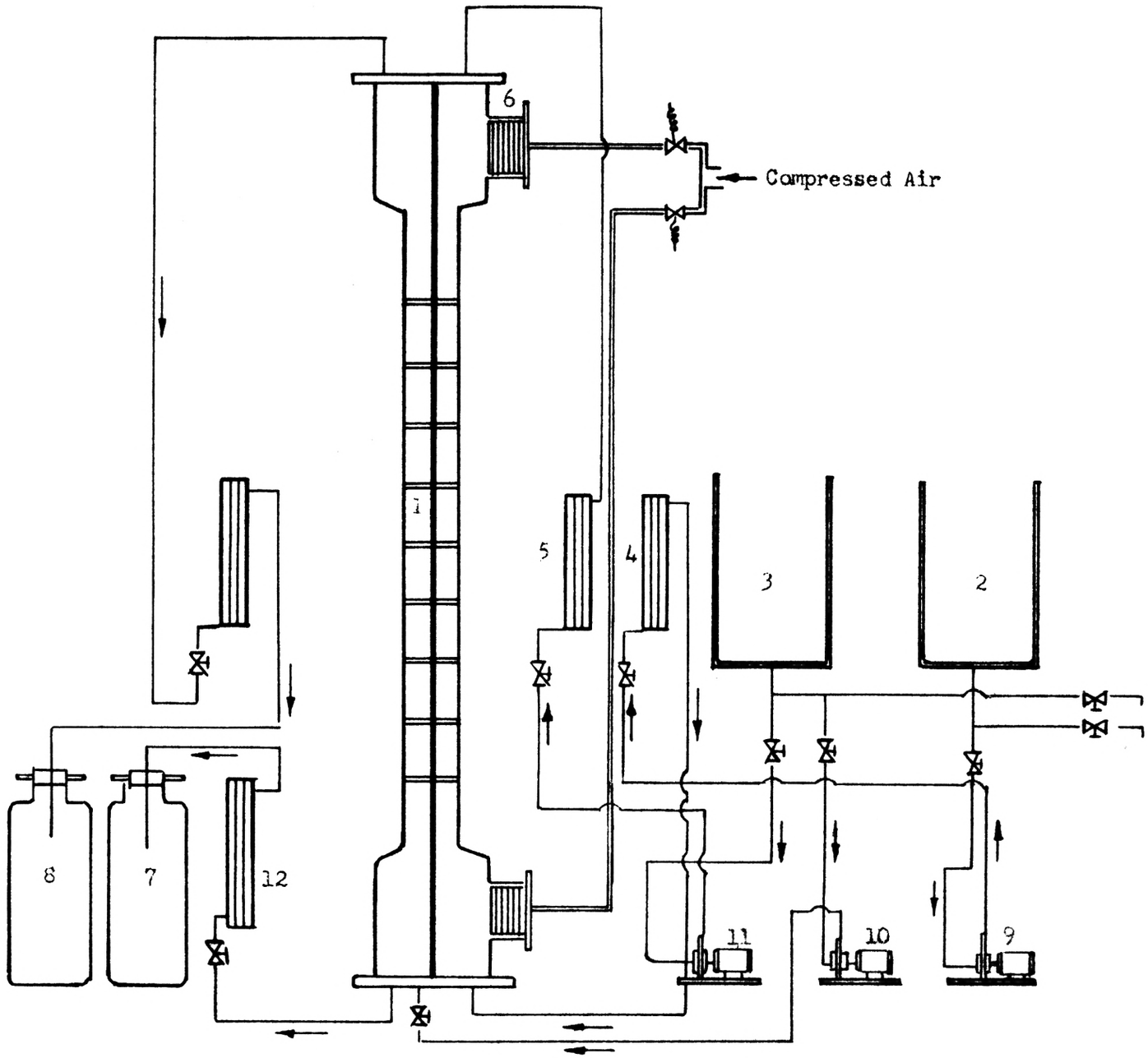
in a Pulse Column

1. Pulse Column
2. Toluene Tank
3. Water Tank
4. Flowrator for Lighter Solvent
5. Flowrator for Feed Solution
6. Teflon Bellows
7. Water Receiver
8. Toluene Receiver
9. Light Solvent Centrifugal Pump
10. Heavier Solvent Centrifugal Pump only used for starting up of operation
11. Heavier Solvent Centrifugal Pump
12. Flowrator for Control of the Interface

PLATE VIII

Solenoid Valve

Needle Valve



or three times the volume of aqueous solution which was originally in the column at start-up had been removed. The method of controlling interface during operation required careful regulation of entering and leaving streams. The frequency and amplitude controls were adjusted to give desired conditions of operation.

Six samples were taken for each specific run. The samples were taken from the toluene tank, from the water solution tank, from the water receiver (after equilibrium) and from the toluene or raffinate receiver. Two samples were taken at the end of the operation from the top and bottom of the column. In order to test for equilibrium conditions in the tower two additional samples were taken at the top and bottom after one-half hour following shut-down. Comparison was made of these two samples measured by titration for benzoic acid content with those taken at shut-down. If both samples had the same concentration, it was assumed that equilibrium conditions had been established in the column.

Preparation of Aqueous Solutions of Benzoic Acid

The 0.001 N aqueous solution of benzoic acid was prepared by dissolving a weighed amount of benzoic acid in hot water, then diluting with distilled water to the desired volume.

Calibration of Column

It was necessary to know the volume of toluene phase and water phase held up in the column. The calibration of the column was done by the simple procedure of filling the column and drawing samples and marking the level within the column with suitable marks.

Calibration of Frequency Timer

During various runs different frequencies were used. By turning the cycle switch on the timer, the calibration of the frequency for different positions of the switch was done by checking with a stop watch. This data is shown in Table 2.

Pulsing Amplitude

The pulsing displacement was controlled by air pressure. For example, higher air pressures produced greater pulsing amplitudes. The calibration was carried out by adjusting the compressed air valve to the air pressure regulator, turning on the timer switch and measuring the linear displacement of the interface level as it traveled up and down in the column.

Method of Analysis to Determine Concentration of Samples

Standard solutions of sodium hydroxide were used in the analysis of the feed and the products as in the determination of equilibrium data. Two types of samples were tested, i.e., aqueous solutions of benzoic acid and toluene solutions of benzoic acid. The former were easily determined by direct titration. The latter were titrated by pipeting 10 cc samples into 250 cc beakers and then adding 5 cc absolute ethyl alcohol and two drops of phenolphthalein indicator. Standard base was used in the titration. A check on the benzoic acid in the recovered toluene was maintained.

GENERAL PERFORMANCE CHARACTERISTICS OF PULSE COLUMN AND ITS VARIABLES

Three different types of phase dispersion behavior have been observed in this pulse column. These are a function of the through-put rate and the pulsing conditions.

Table 2. Calibration of frequency timer

Number	:	Pulsation/minute
Frequency for bottom bellows; off cycle (on cycle set on 100)		
100		5.6
90		5.6
80		5.7
70		6.0
60		6.3
50		6.7
40		7.1
30		7.5
20		7.6
10		7.0
0		7.0
Frequency for top bellows; on cycle (off cycle set on 100)		
100		5.5
90		5.6
80		6.0
70		6.5
60		7.2
50		7.7
40		8.65
30		10.0
20		10.0
10		11.6
0		

Frequent checks on the amplitude were necessary from time to time because of slight changes in air pressure and pump inputs of feed and solvent.

Mixer-Settler Type Operation

This type of operation occurs at low through-put rates and low frequencies and low amplitudes. It is characterized by the separation of the light and heavy phase into clear layers in the interplate space during operation. When the bottom bellows pushes the solution upward, the light phase immediately resting under a sieve plate will be forced up through the perforated plates and will rise as small globules through the heavy phase layer above the plate. Similarly, the heavy phase will descend during downward movement of the pulse. This type of operation is highly stable.

Emulsion Type Operation

This type of operation occurs at higher through-put rates and frequencies and amplitudes. It is characterized by small drop size (typically about 1/32-inch in diameter or larger) and fairly uniform dispersion during operation. It has high interfacial contact area per unit volume.

Unstable Operation

This type of operation (14) occurs at still higher through-put rates and frequency and amplitude. It is characterized by mixtures of fine and coarse dispersed drops. It forms large irregular shaped globules of dispersed phase by coalescence. The efficiency of the column in unstable operation is generally lower.

As the through-put rate and the frequency are increased still further, beyond the unstable region, complete flooding results.

Summation of Variables

The main factors which affect performance may be grouped as follows:

a. Pulse amplitude, frequency and through-put rate.

b. Phase flow ratio, choice of continuous phase.

c. Plate geometry including hole size, plate spacing, percentage of free area, and column diameter. In this research these were fixed.

d. Component concentration.

A picture of the appearance of the pulsing operation is shown in Plate IX.

CALCULATION OF DATA

A convenient method for estimating the height of a counter current extraction column is based on the concept of the height of a transfer unit (H.T.U.) which was introduced originally by Chilton and Colburn (Bigford, et al., 13). This method is direct and rapid especially for rough estimates. The mass transfer characteristics of the packing is expressed in terms of an experimentally determined number which has the dimensions of length only. The basic concept is that the estimation of the height of the column always depends upon the evaluation of a definite integral, such as

$$\text{H.T.N.} = \int_{y_2}^{y_1} \left(\frac{dy}{y - y_e} \right)$$

where y_e = mole fraction of solute in water phase at equilibrium with bulk of organic solvent

y_1 = mole fraction of solute in the aqueous stream at the concentrated end of the counter current apparatus

y_2 = mole fraction of solute in the aqueous stream at the dilute end of the counter current apparatus

H.T.N. = number of overall aqueous phase transfer units

EXPLANATION OF PLATE IX

View of Dispersed Toluene Phase Through Aqueous Phase in Mixer-Settler Type Operation

Fig. 1 Observe the whiteness in liquid portion during the upward stroke. This is the result of fine or very small toluene droplets in the water phase.

Fig. 2 Relative clearness of the liquid portion in Fig. 2 as compared with that in Fig. 1 served to illustrate less dispersion of toluene droplets on the downward stroke.

PLATE IX

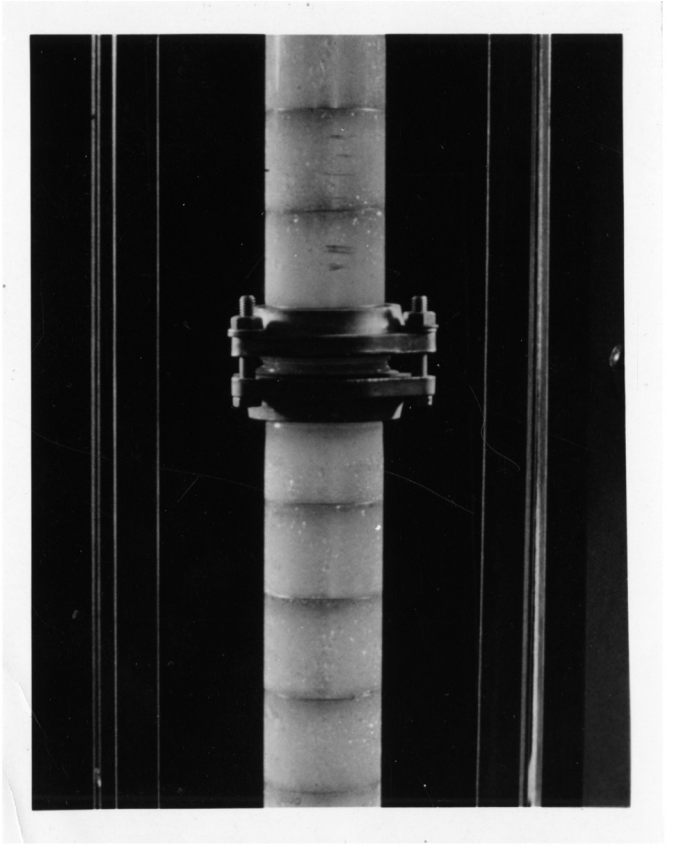


Figure 1.

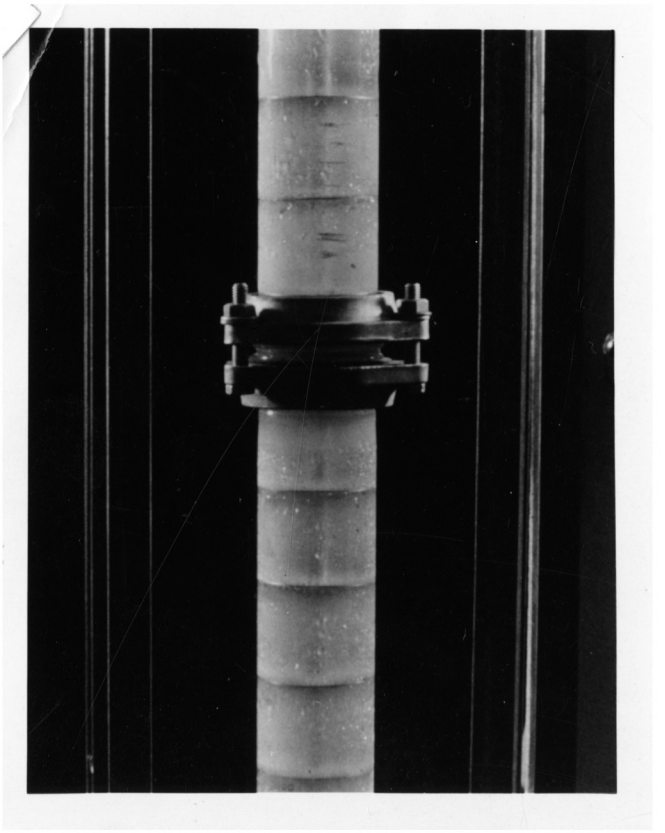


Figure 2.

The value of this integral, which is dimensionless, expresses the difficulty of extracting the solute from the water solution. It is called the "Number of a Transfer Unit" and is based on the overall aqueous phase driving force, i.e., the smaller the driving force, the greater the H.T.N.

The equation, used for calculating the column height for the case of dilute aqueous streams, is:

$$h = (H.T.N.)(H.T.U.)$$

where h = height of the column.

H.T.U. = height of an overall water-phase transfer unit, ft.

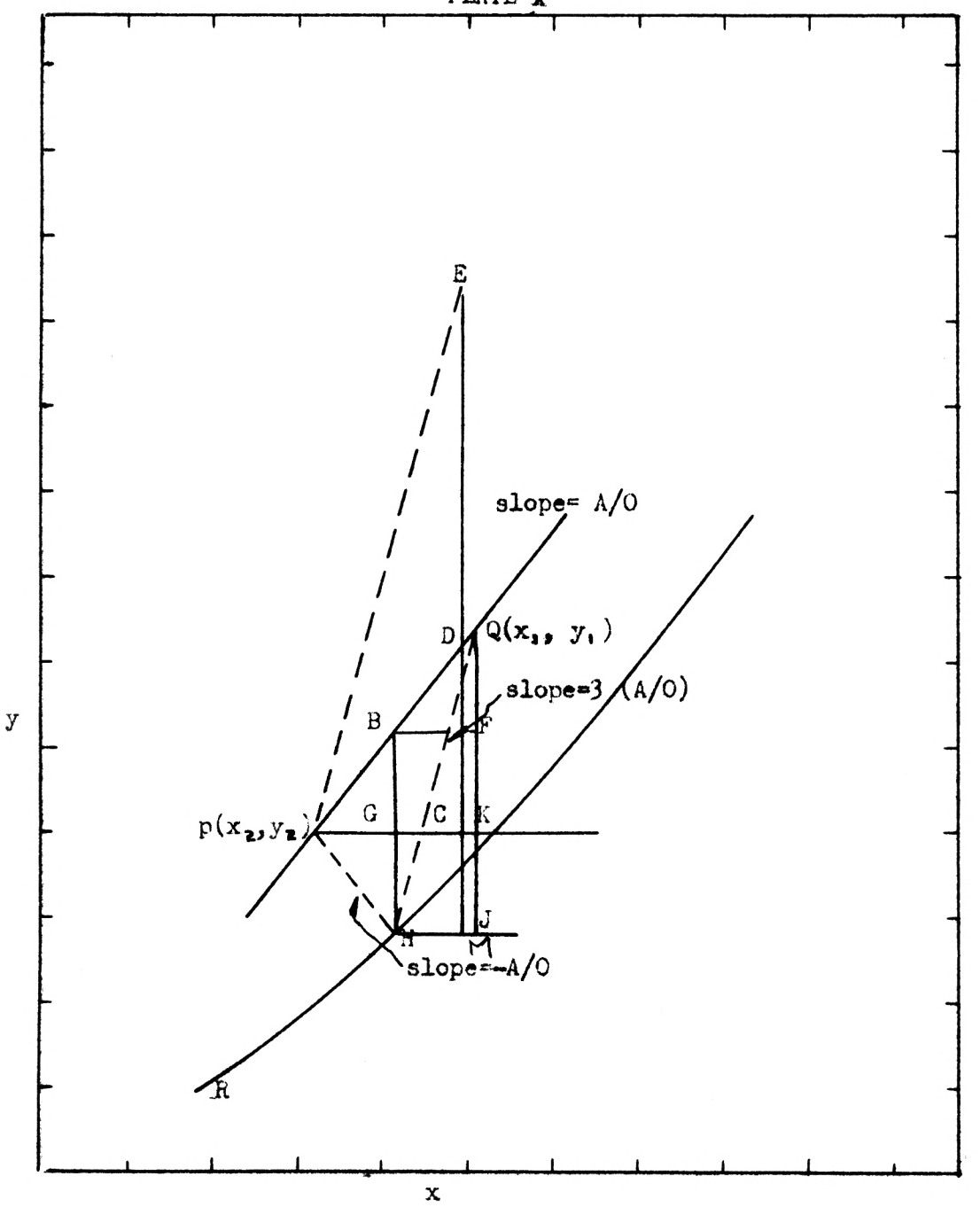
Before calculating the H.T.U., it is necessary to calculate the H.T.N.

The method used here to calculate the H.T.N. is the graphical construction method suggested by White (Pigford, et al. 13). The principle of this calculation is illustrated first by applying it to the case as shown in Plate X. Suppose RH is an equilibrium curve and PBD is the operating line whose slope is A/O. Starting at point P (x_1, y_1) on the operating line, a horizontal line is drawn. Then any vertical line, ME, is drawn intersecting this horizontal line at C to the right of P. Line ME intersects the operating line at point D. Points M and E are located so that CE = 3CD and M = DC. Another line will be drawn through point P on the operating line with slope -A/O and which intersects the equilibrium curve at point H. Finally a line is drawn through H parallel to PE which intersects the operating line at Q (x_2, y_2). This completes one step for the evaluation of one H.T.N. The process is continued until the terminal value has been reached. The validity of this construction is proved if BH can be shown to equal both the mean driving force and the change in y, i.e., ($y_2 - y_1$). Triangle PHG is equal to triangle PGB, therefore, BG is equal to BH. Since HQ has three times the slope of BQ, QJ equals 3QF and QF equals BG. Since QK equals BH equals

EXPLANATION OF PLATE X

Principles of graphical determination of the height transfer number by method of White

PLATE X



$(y_2 - y_1)$, the construction is proved. This method has some advantage to be used for stepping off transfer units in either direction.

The procedure for the calculation of H.T.N. for the extraction of benzoic acid by toluene is summarized as follows (Plate XI):

1. Locate a point P_2 (x_2, concentration of feed and y_2, concentration of raffinate) and point P_1 (x_1, concentration of extract and y_1, concentration of solvent) which determine the operating line.

2. The slope of this operating line is checked to compare it with ratio of feed to solvent, A/0, which was measured during operation.

3. Select any point P_3 on the operating line. Draw P_3Q and P_3R so that slope of P_3Q will be equal to 1/3 x A/0 and P_3R equal to -A/0.

4. The equilibrium curve will be selected for the corresponding temperature during operation.

5. Starting from P_1, draw line P_1A parallel to P_3R. It will intersect the equilibrium curve at A. Then through A, another line, AB, is drawn parallel to P_3Q which will intersect on the operating line at B, and so on. The H.T.N. obtained as result of this is equal to 2.2.

Then from the equation

$$h = (H.T.U.)(H.T.N.)$$

where h = height of pulse column in feet, H.T.U. in feet was calculated.

INTERPRETATION OF DATA

Effect of Pulse Amplitude and Frequency

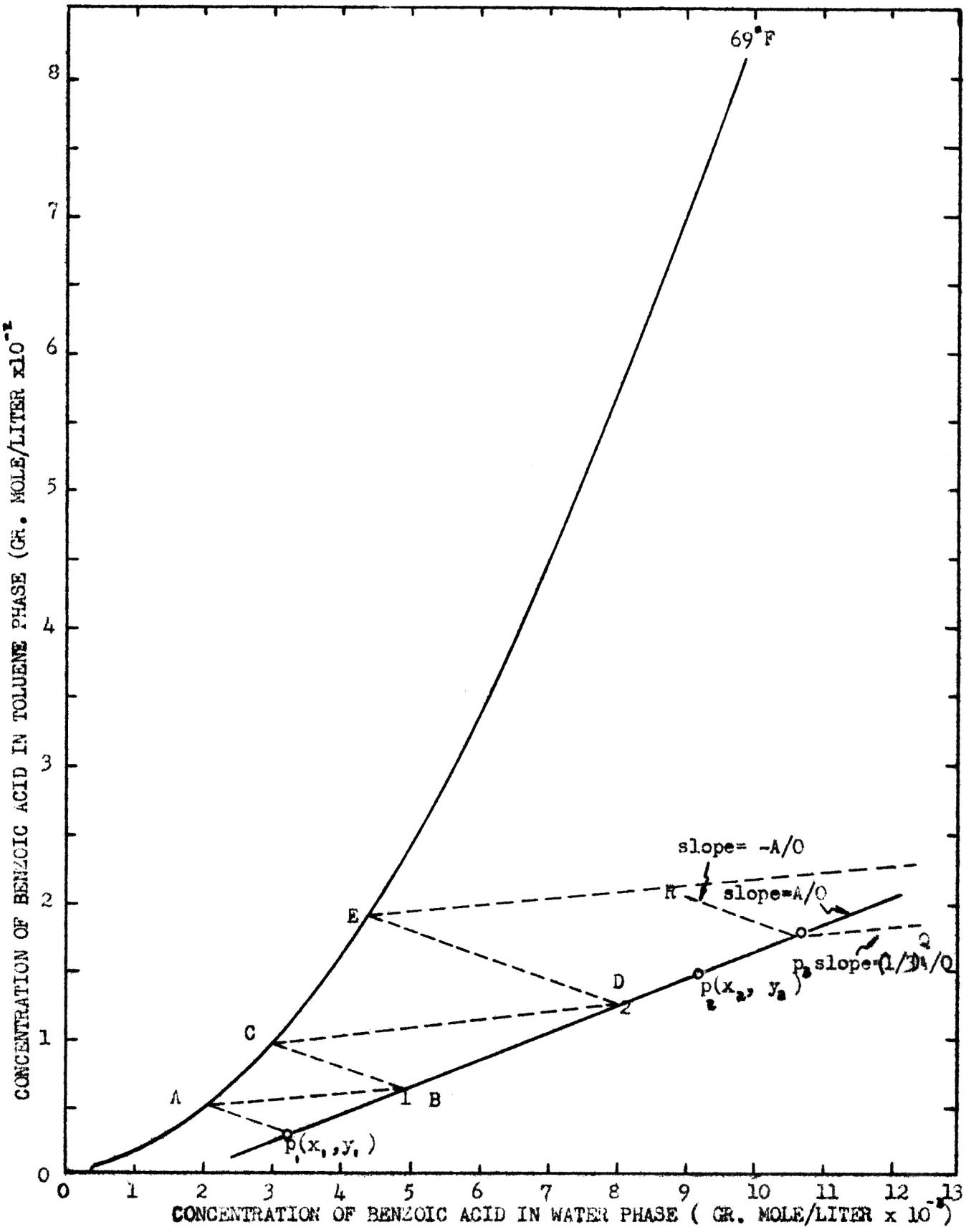
The curve on Plate XII shows the relationship between H.T.U. and amplitude at various values of frequency. The trend of the curves shows a sharp increase in H.T.U. and then a slow decrease with increasing amplitudes. A

EXPLANATION OF PLATE XI

Graphical determination of the height transfer number (H.T.N.) by method of White

H.T.U. = 2.2 in this example

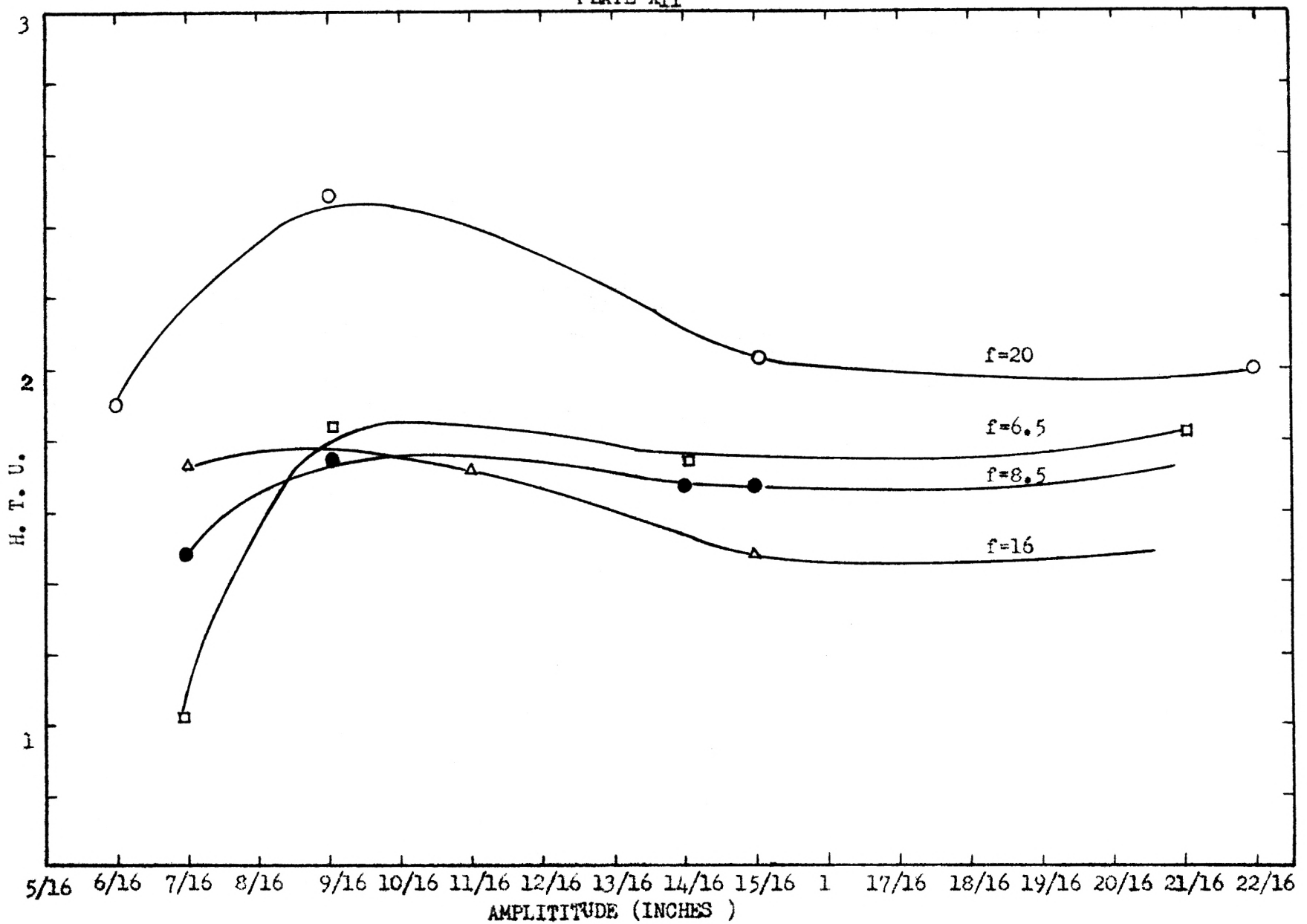
PLATE XI


69°F
slope= -A/O
slope=A/O
slope=(1/3)A/O
R
E
D
C
A
B
1
2
3
$p_1(x_1, y_1)$
$p_2(x_2, y_2)$
p_3
CONCENTRATION OF BENZOIC ACID IN TOLUENE PHASE (GR. MOLE/LITER x10^{-2})
CONCENTRATION OF BENZOIC ACID IN WATER PHASE (GR. MOLE/LITER x 10^{-5})
0
1
2
3
4
5
6
7
8
9
10
11
12
13

EXPLANATION OF PLATE XII

Plot of H.T.U. and amplitude at various values of frequency at V (through-put rate) = 120 cc/min. A/0 (phase flow ratio) = 2

PLATE XII


3
2
1
H. T. U.
f=20
f=6.5
f=8.5
f=16
5/16 6/16 7/16 8/16 9/16 10/16 11/16 12/16 13/16 14/16 15/16 1 17/16 18/16 19/16 20/16 21/16 22/16
AMPLITITUDE (INCHES)

further slight increase of H.T.U. occurred. The maximum point of these curves occurred at an amplitude range from 9/16 to 11/16 inches. For amplitudes up to 11/16 inches the operation was in mixer-settler type. There was a gradual transition to emulsion type dispersion when the amplitudes were further increased. That is, H.T.U. decreased with a rise of amplitude.

In order to explain this situation it was necessary to assume that in the mixer-settler type, the dispersion of toluene during pulsation was different than it was in the emulsion type. At low amplitudes there was only a very short displacement of toluene droplets during the downward pulse. This was partially explained by the fact that, since the specific gravity of toluene is less than that of water, the displacement of toluene through the continuous aqueous phase required less energy during pulsation to force the small droplets of toluene to rise through the water phase than that which was necessary to force them back down. Therefore, an increase in the amplitude caused an increase in the movement of toluene bulk through the column and resulted in a decreased chance of contact between the phases. The mass transfer from the water phase to toluene phase was lowered and the H.T.U. value was increased in the mixer-settler type of operation.

With further increase in amplitude, that is in the range of emulsion type operation where larger forces were required to push the toluene through the small holes of the plates, the size of the toluene droplets became smaller and smaller. This was assumed to be the explanation for the existence of emulsion type phase being formed and resulted in the area of contact between both phases being raised. The emulsion appeared to consist of small droplets of toluene surrounded by water films and was designated as a two phase homogeneous mixture. It was, therefore, difficult to separate the phases by settling in the short time intervals allowable between pulses.

Thus the finely divided particles of the mixture were more easily forced downward through plates than they were in mixer-settler type of operation. Therefore, in emulsion type operation, an increase in amplitude produced less increase in the movement of toluene bulk through the column but resulted in an increased interphase area contact, but resulting in a decrease in H.T.U.

The curve on Plate XIII shows the relationship between H.T.U. and frequency at various values of amplitudes. The trend of the curves shows a decrease in H.T.U. with an increase in frequency, followed by an increase of H.T.U. The minimum points of the curves ranged from 11 to 13 cycles per minute. The optimum frequency with regard to H.T.U. occurred during amplitude ranges from 9/16 inches to 19/16 inches. Optimum H.T.U. values were always obtained at the lower frequencies when the values of amplitude were less than 9/16 inches or greater than 19/16 inches. The lowest curve in Plate XIII with an amplitude of 7/16 inches corresponded to mixer-settler type of operation. The 2nd and 3rd curves with amplitudes from 9/16 inches to 19/16 inches corresponded to emulsion type operation.

It was necessary to explain the effect of the values of H.T.U. with an increase in frequency as well as with an increase of amplitude in mixer-settler type operation. An increase in frequency resulted in an increase in the movement of toluene bulk through the column with a resulting increase in H.T.U. values.

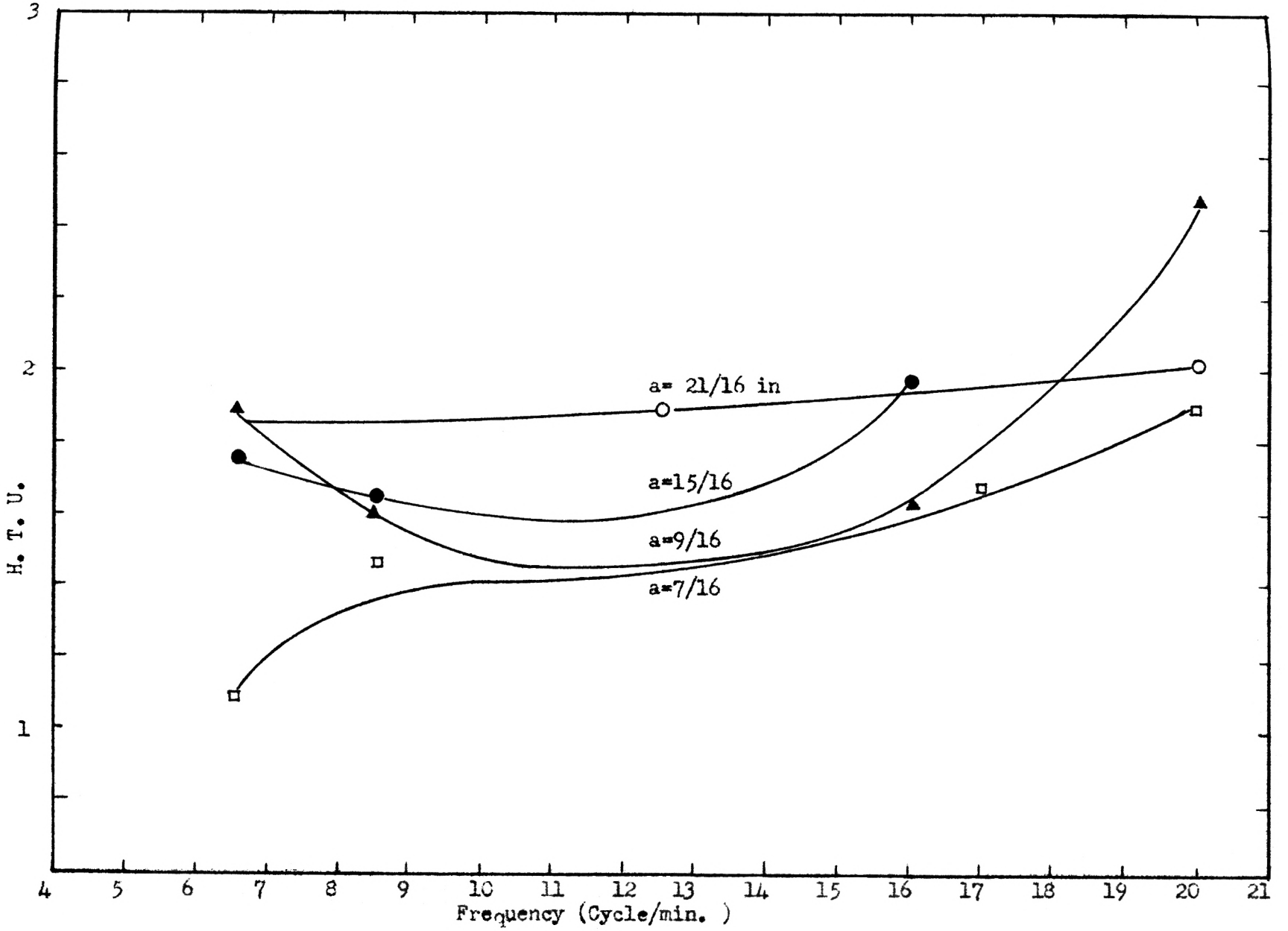
In the case of emulsion type dispersion an increase in frequency produced both an increase in the chance of contact between phases (because of solution being emulsified smaller droplets of toluene will be more readily pushed downward in the column) and in the interphase contact area with a resulting decrease in H.T.U. values until it reached the minimum point of the curves (13 cycles per minute).

EXPLANATION OF PLATE XIII

Plot of H.T.U. versus frequency at various values of amplitude at V (through-put rate) = 120/min.

A/0 (phase flow ratio) = 2

PLATE XIII


3
2
1
H. T. U.
a= 21/16 in
a=15/16
a=9/16
a=7/16
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
Frequency (Cycle/min.)

If the frequency was further increased, the column would be operated close to an unstable or a flooding condition. In this case it was difficult for the toluene droplets to separate from aqueous phase by their own gravity in the short time interval allowable and sometimes they would come out with the aqueous extract. Therefore, poor effective mass transfer between phases in such a situation was encountered. In the case of higher amplitude, 21/16 inches, the operation was in the flooding stage and resulting poor operation, i.e., high H.T.U. values.

Effect of Through-Put Rate

The H.T.U. values were relatively insensitive to variations in the through-put rate, especially at the low values. In Plate XIV H.T.U. values were plotted against various values of through-put rate, expressed in terms of the sum of the superficial pulse capacity of the two phases. The units of pulse capacity were expressed in gallons per square foot per hour which are discussed below. These curves are nearly straight lines indicating that H.T.U. values closely approximated the optimum value over a wide range and with rising H.T.U. values at higher ratios.

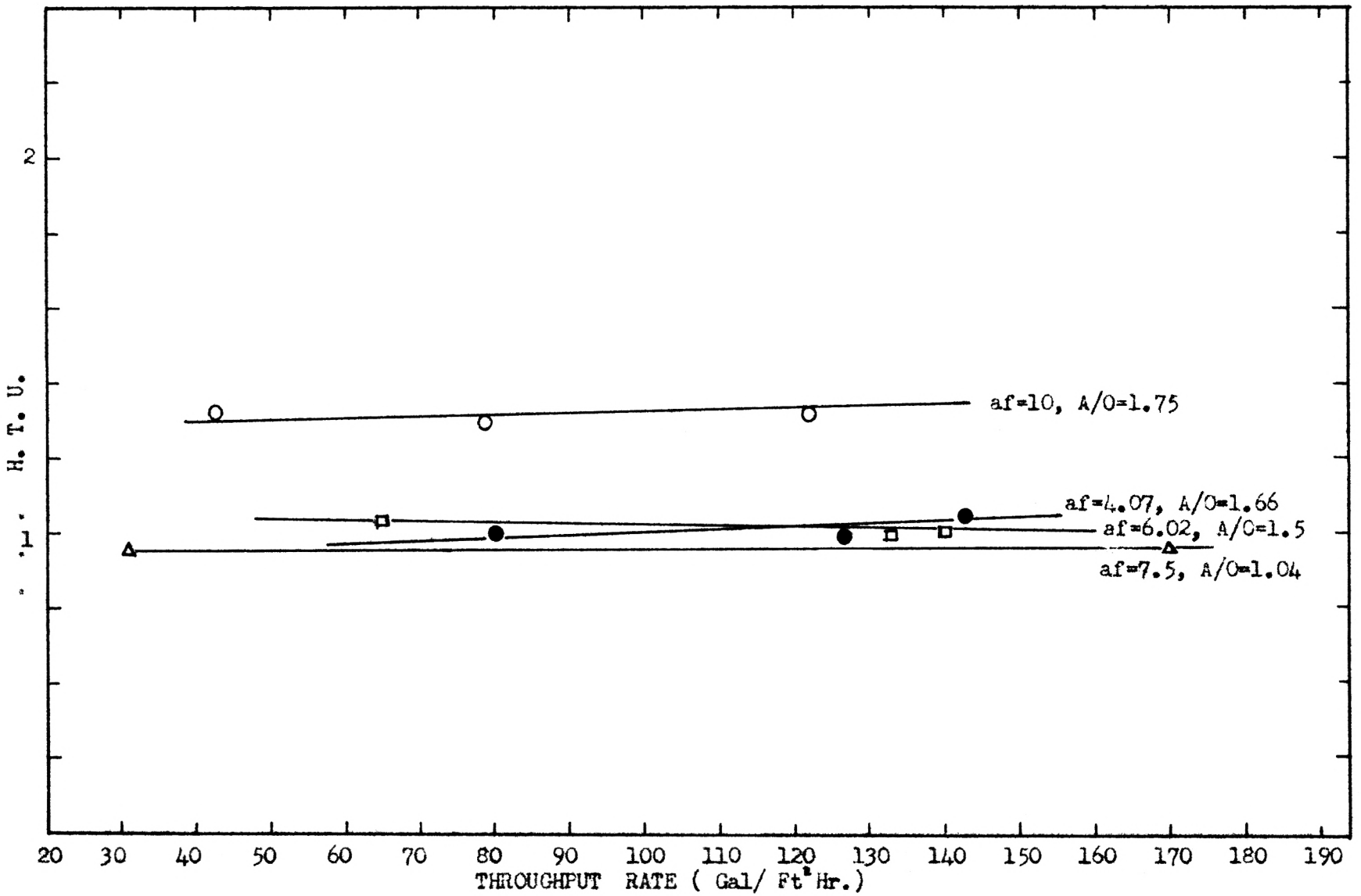
Effect of Phase Flow Ratio

The aqueous phase has been chosen as the continuous phase. Plate XV shows that H.T.U. values increased with an increasing ratio of the continuous to dispersed or toluene flow. An increase in the dispersed phase flow resulted in an increase in interphase contact area. Usually from the standpoint of extraction effectiveness, it was advantageous to make the continuous phase with the smaller flow and the dispersed phase with the larger flow, i.e., lower H.T.U. values. At low ranges of phase flow ratio

EXPLANATION OF PLATE XIV

Plot of H.T.U. versus through-put rate at various values of af and A/0

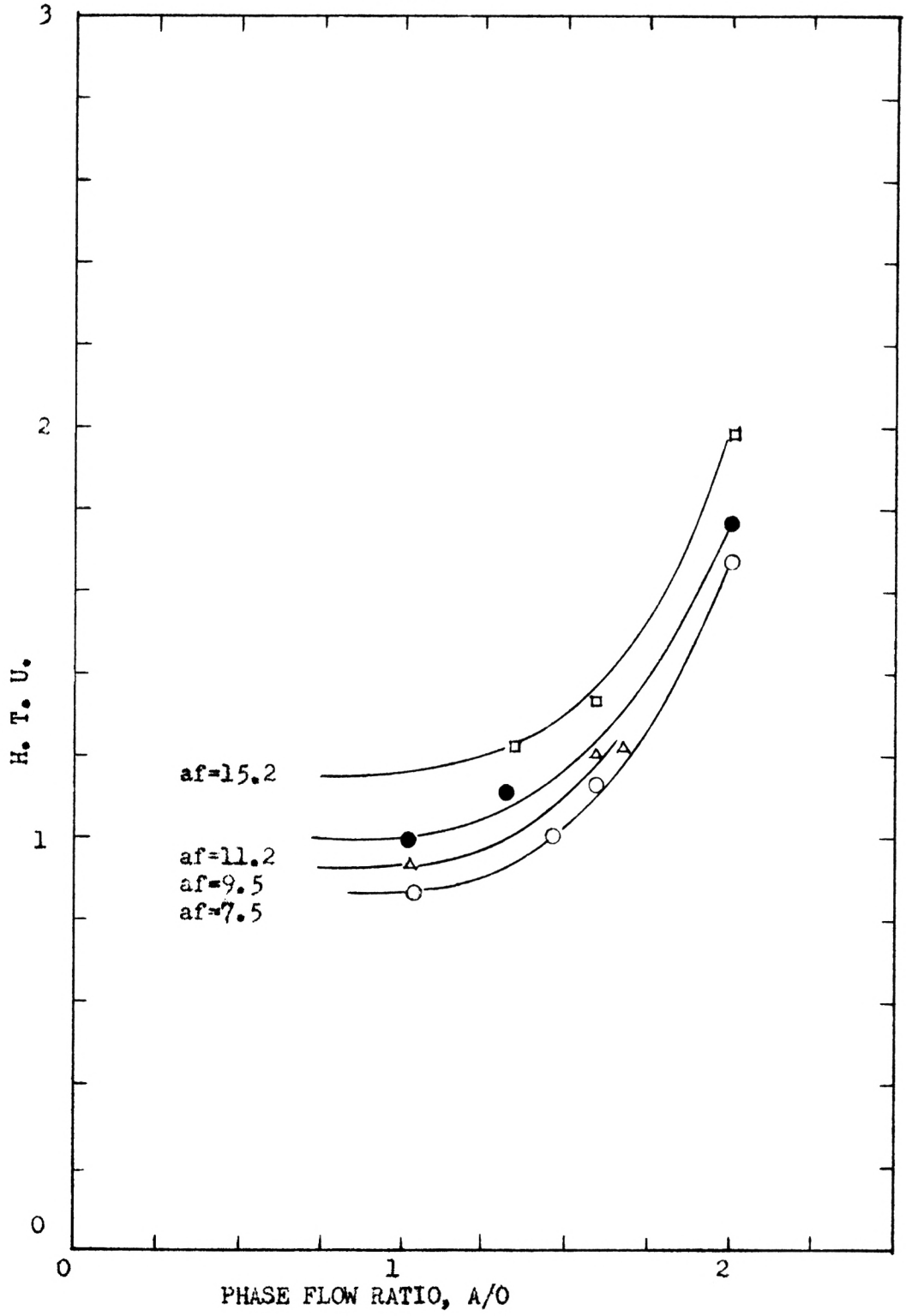
PLATE XIV


2
1
H. T. U.
af=10, A/O=1.75
af=4.07, A/O=1.66
af=6.02, A/O=1.5
af=7.5, A/O=1.04
20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190
THROUGHPUT RATE (Gal/ Ft² Hr.)

EXPLANATION OF PLATE XV

Plot of H.T.U. versus phase flow ratio at various values of af

PLATE XV



H.T.U. increased very slowly with increasing values of A/O. After a value of 1.5 for A/O was reached, H.T.U. rose rapidly and there seemed to be a tendency for the curves to come closer together as the flow phase ratio further increased. This could be interpreted to mean that there would be no effect of displacement of liquid in the pulse volume at high phase flow ratios. In order to explain this phenomenon, it is necessary to assume that, in the case of using a larger quantity of aqueous continuous phase with relatively smaller quantities of the dispersed phase, the change in c_A, the concentration of benzoic acid in the aqueous feed, would be small and c_t, the concentration in toluene, would remain approximately constant. This would indicate a constant H.T.U. at high phase flow ratio, and would be independent of optimum amplitude and frequency. H.T.U. values would approach infinity with further increase of the ratio A/O. Therefore, there would be an asymptote of those curves which would also be the limit of phase flow ratio.

Relationship Between Pulse Amplitude, Frequency and Flow Ratio

The physical significance of the relationship between H.T.U., phase flow ratio and the product of amplitude and frequency showed clearly in this investigation.

The term amplitude "a" was used to indicate the magnitude of pulse displacement of interface from one extreme position to the other in the column with a corresponding frequency. In order to understand the physical significance of the meaning of the product (a)(f), the term "pulse capacity" was introduced and was defined in units such as gallons pulsed per hour per square foot of column cross section. It was calculated by multiplying the pulse frequency, (f) (in cycles per hour), by the amplitude (a) in gallons

(employing the sum of the up and down displacement) and then dividing by column cross section in square feet. It was directly proportional to both the pulse amplitude (a) and frequency (f).

The curves on Plate XVI show the relationship between H.T.U. and (a)(f) at various values of phase flow ratio. The optimum condition was always at low values of (a)(f). The value of H.T.U. increased rapidly with an increase of (a)(f) in the range of 3 to 7, then increased very slowly thereafter. This means that there was only small effect on the value of H.T.U. by changing the pulsed capacity. Therefore an assumption was made that, in the region low values of (a)(f) an increase of pulse capacity resulted in an increase of movement of dispersed phase passing through the continuous phase. At high values of (a)(f) the effect of an increase in interphase area was approximately cancelled out.

Mathematical Equations of Correlations

In order to find the mathematical correlation of the values of H.T.U. the product of frequency and amplitude (pulsed displacement) and flow phase ratio, the data were plotted on coordinate paper to show the relationship between those variables. When $(H.T.U.)^{\frac{1}{2}}$ was plotted as ordinates versus (a)(f) as abscissa, a group of parallel straight lines was obtained (Plate XVII).

The mathematical expression is shown by the equation

$$(H.T.U.)^{\frac{1}{2}} = \phi \left[A/O,\ (a)(f) \right]$$

where A = phase flow rate of aqueous solution

O = phase flow rate of toluene

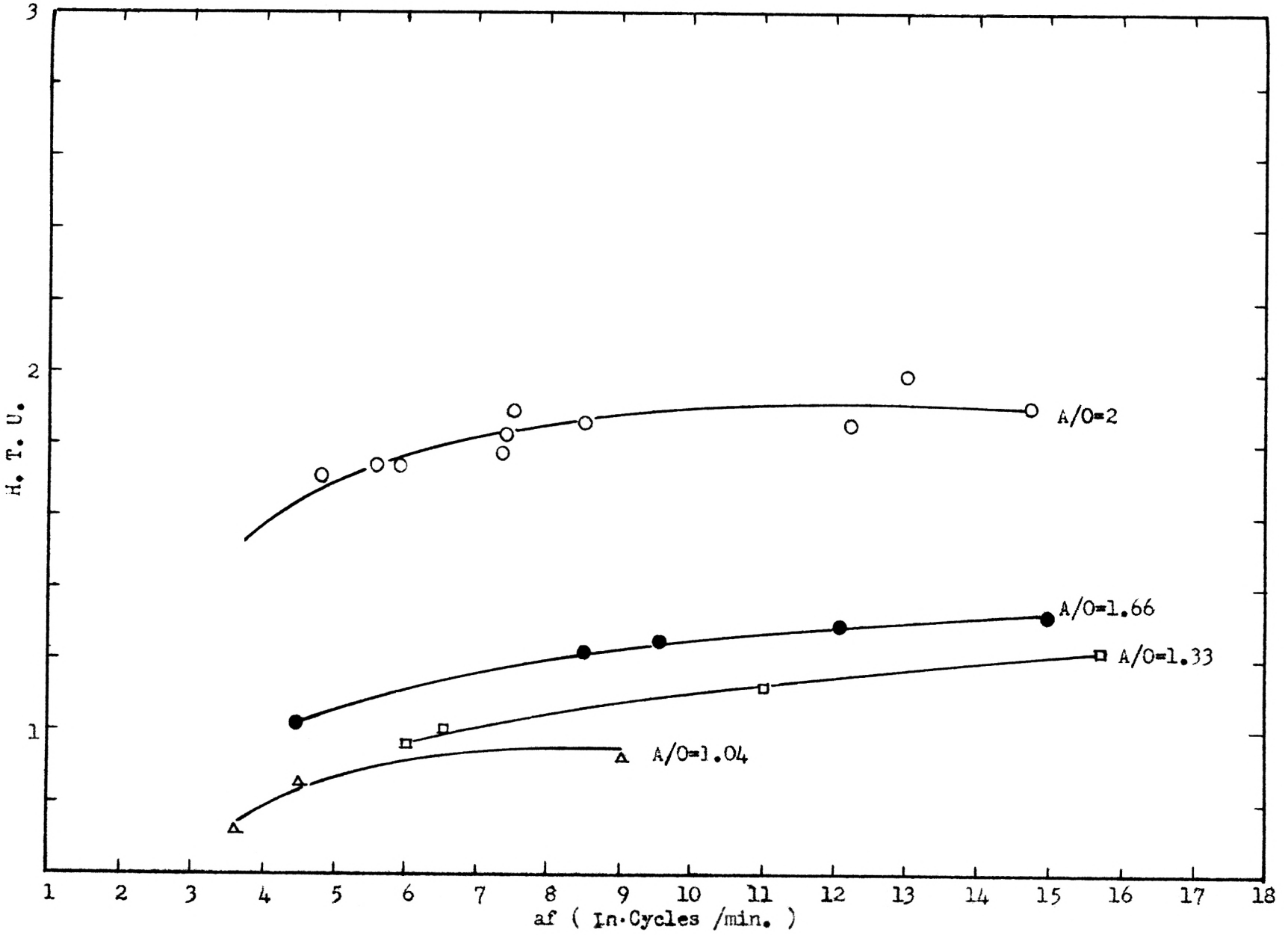
a = amplitude (pulsed displacement in inches)

f = frequency (cycles/minute)

EXPLANATION OF PLATE XVI

Plot of H.T.U. versus product of frequency and amplitude at various values of phase flow ratio, A/O

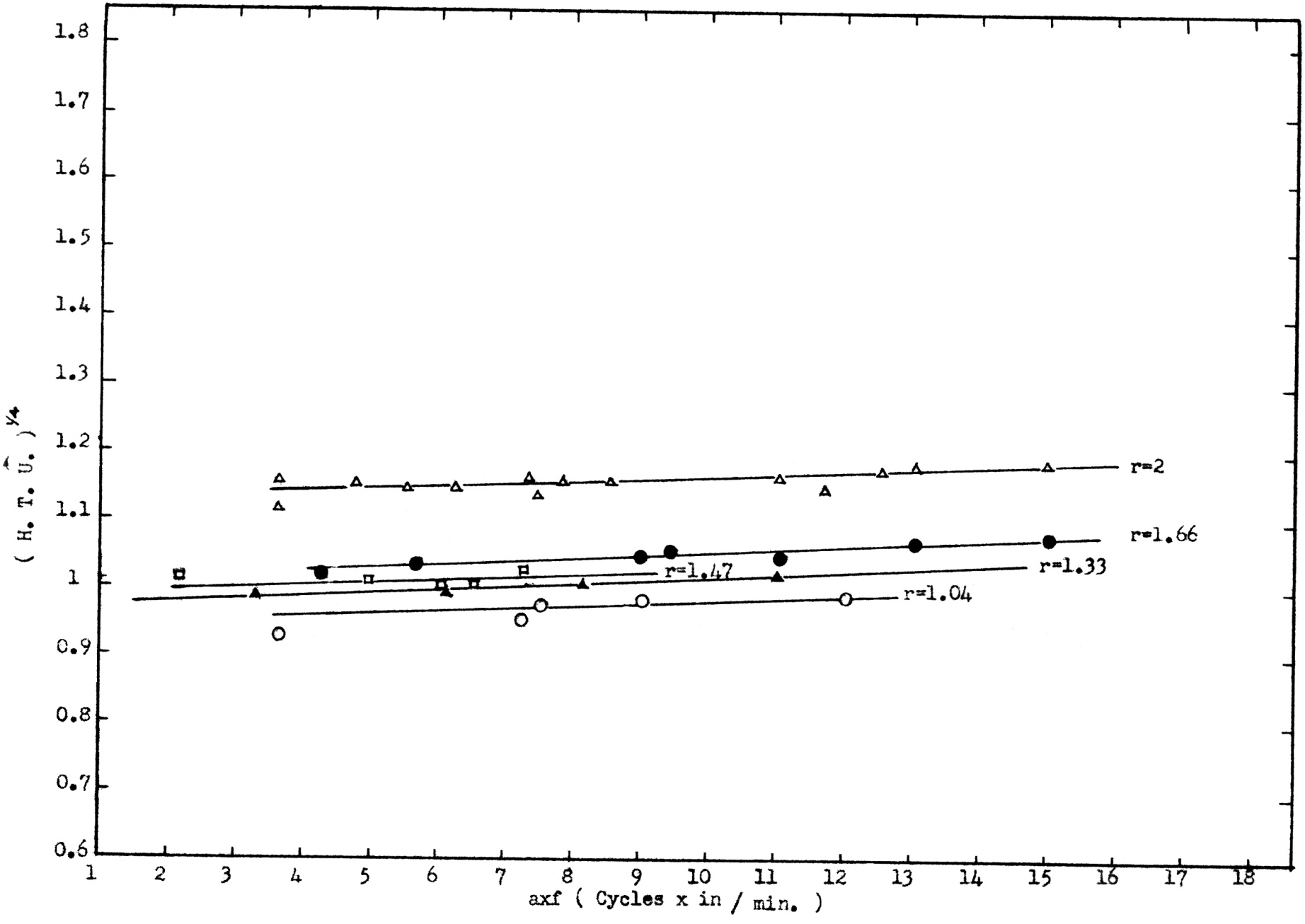
PLATE XVI



EXPLANATION OF PLATE XVII

Plot of $(H.T.U.)^{\frac{1}{4}}$ versus product of amplitude and frequency at various values of phase flow ratio, A/O

PLATE XVII


1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
0.6
(H. T. U.)^1/4
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
axf (Cycles x in / min.)
r=2
r=1.66
r=1.47
r=1.33
r=1.04

A detailed analysis of the mathematical equations for the relationship between H.T.U. and different phase rate ratios follows: In the equation

$$(H.T.U.)^{\frac{1}{4}} = L_n\ (a)(f) + C_n$$

L is the slope of those lines, and C is the intercept, which is not constant, and it is also a function of A/0. Evaluation of constants is shown in Table 3.

Table 3. Constants of equation.

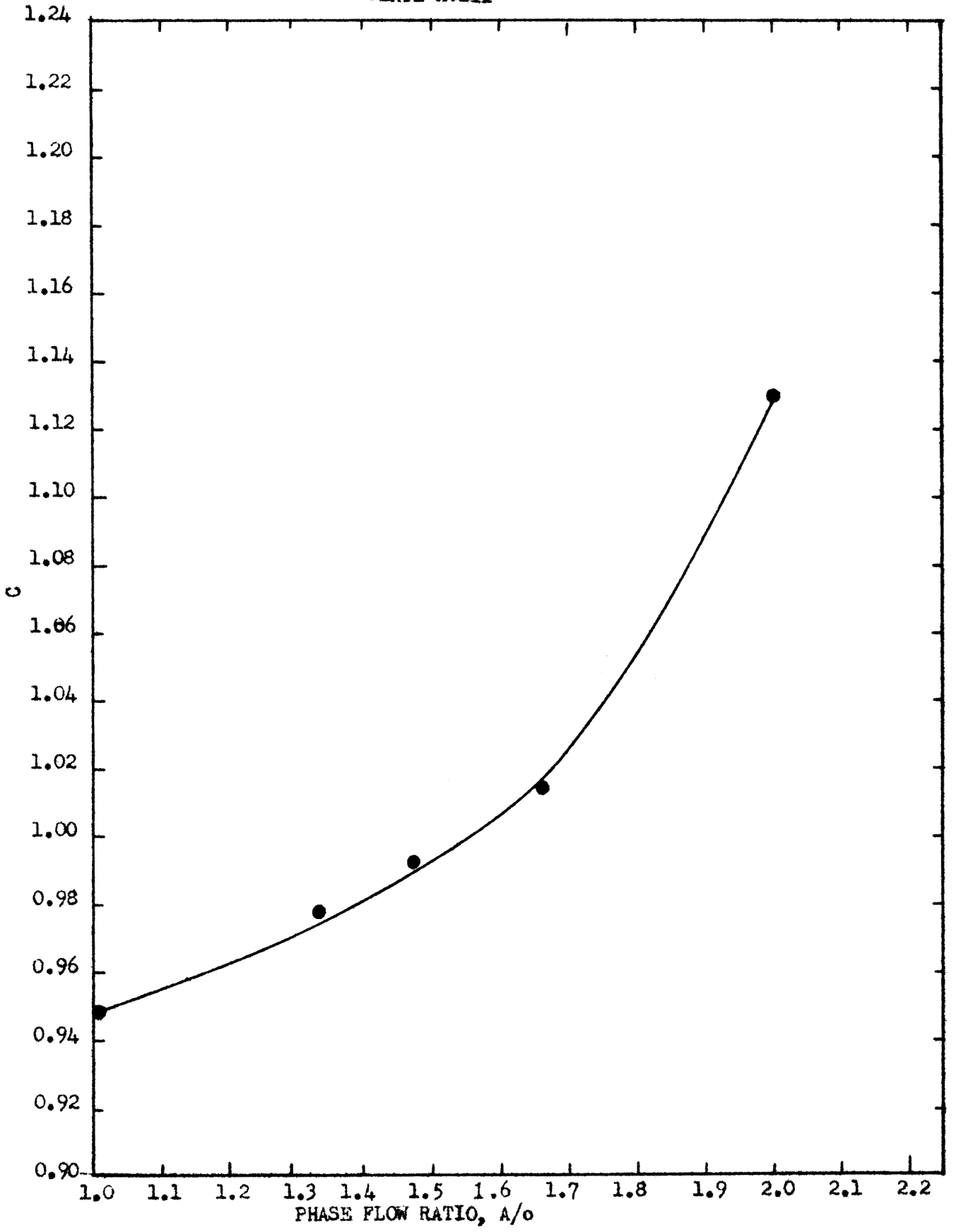
A/0	:	C_n	:	L_n
2		1.130		0.00400
1.66		1.015		0.00405
1.47		0.993		0.00385
1.33		0.978		0.00370
1.04		0.956		0.00400

In order to simplify the equation an average value of L = 0.004 was used for the different values of flow ratio. In order to find the relationship between A/0 and C a curve was plotted with C as ordinate and A/0 as abscissa (Plate XVIII). A smooth curve was obtained and C, therefore, can be expressed by function $C = (a)(A/0)^n$ where C and A/0 are variables; "a" and "n" are constants which have to be determined. When A/0 equals to zero, C is equal to 0.945. Log (C-0.945) was plotted versus log A/0 as shown on Plate XIX and is a straight line. The calculations are shown on the following table.

EXPLANATION OF PLATE XVIII

Plot of phase flow ratio, A/O, versus C

PLATE XVIII


1.24
1.22
1.20
1.18
1.16
1.14
1.12
1.10
1.08
1.06
1.04
1.02
1.00
0.98
0.96
0.94
0.92
0.90
c
1.0
1.1
1.2
1.3
1.4
1.5
1.6
1.7
1.8
1.9
2.0
2.1
2.2
PHASE FLOW RATIO, A/o

EXPLANATION OF PLATE XIX

Plot of log (c-0.945) versus log A/0 for determination of both values "a" and "n"

PLATE XIX

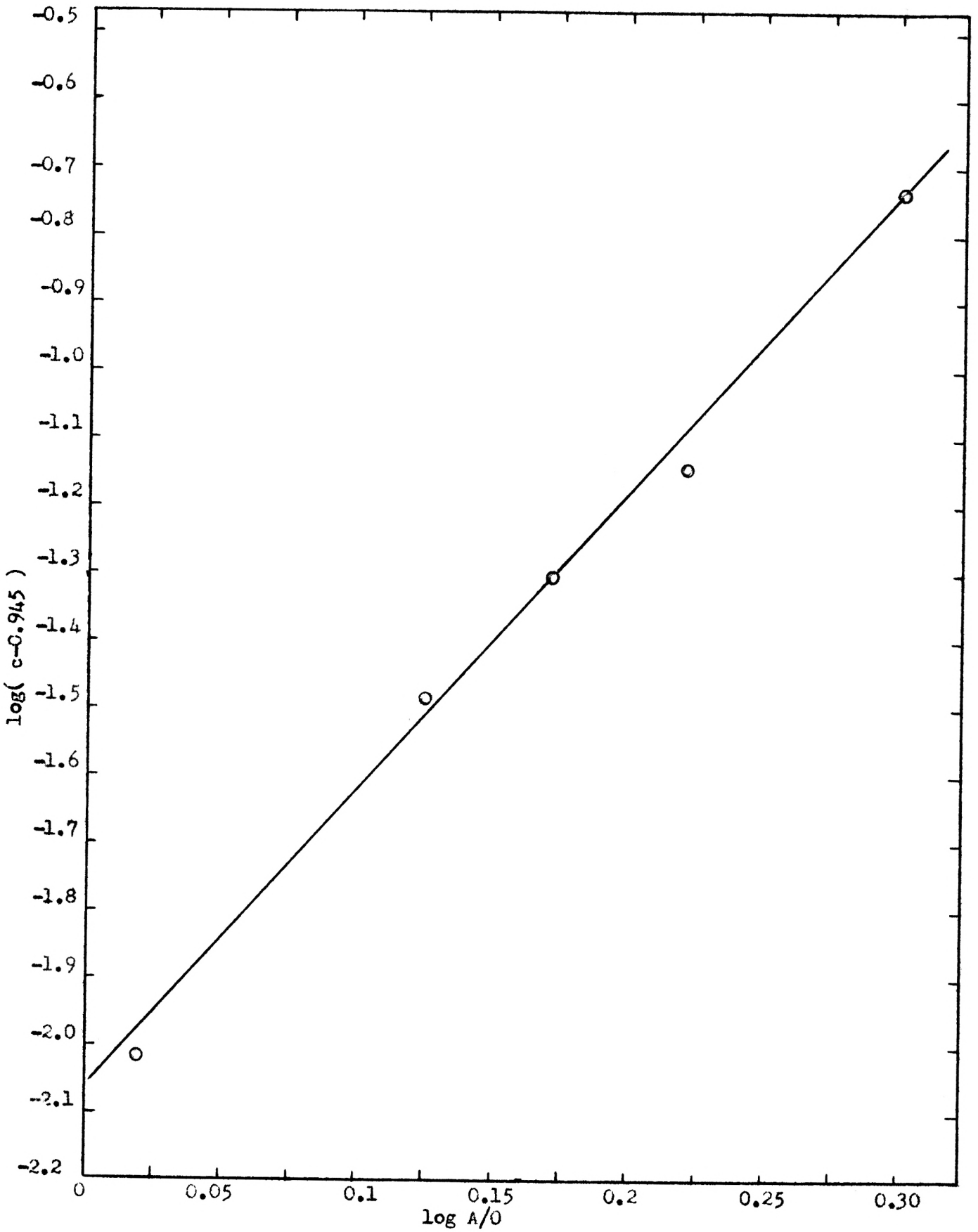

-0.5
-0.6
-0.7
-0.8
-0.9
-1.0
-1.1
-1.2
-1.3
-1.4
-1.5
-1.6
-1.7
-1.8
-1.9
-2.0
-2.1
-2.2
log(c-0.945)
0
0.05
0.1
0.15
0.2
0.25
0.30
log A/0

Table 4. Calculations of log A/0 and log (C-0.945).

A/0	2	1.66	1.47	1.33	1.04
C	1.13	1.015	0.993	0.978	0.952
log A/0	0.3010	0.220	0.1673	0.1239	0.017
C-0.945	0.185	0.07	0.049	0.033	0.0090
log(C-0.945)	0.7328	-1.1549	-1.3187	-1.4815	-2.0458

The equation of the line is

$$C = 0.945 + (a)(A/0)^n$$

which was assumed to be a satisfactory representation of the data, provided that after evaluation of the constants the discrepancies between calculated and observed values are no greater than the assumed error in the experimental determinations. The constants "a" and "n" were taken from Plate XIX, where "n" will equal the slope of the line which value is 4.4, and log a, the intercept was found to be -2.06. Therefore,

$$\log (C-0.945) = 4.4 \log A/0 - 2.06$$

$$\text{and } \log (-0.945) = \log(A/0)^{4.4} + \log 0.00871$$

$$\therefore C-0.945 = 0.00871 \ (A/0)^{4.4}.$$

These results were tested by comparing the calculated and observed value of "C" for each value of A/0 in Table 5.

Table 5. Calculation of R and R^2

A/0	C	Ccal	$R^{x10^{-2}}$	$R^2 \times 10^{-4}$
2	1.130	1.127	-0.03	0.09
1.66	1.015	1.017	0.20	0.04
1.47	0.993	0.9925	-0.05	0.0025
1.33	0.978	0.9756	-0.24	0.0576
1.04	0.952	0.9556	0.36	0.1296

$$\Sigma R = -0.03 \quad \Sigma R^2 = 0.3197$$

The last two columns gave the values of R and R^2, where R was the difference between the calculated and observed values of C. The maximum error R was 0.0036 and Σ R was -0.0003 and ΣR^2 was a better indication of the validity of the equation. Therefore, the equation of correlation can be written as follows:

$$\text{H.T.U.} = \left[0.004\ (a)(f) + C\right]^4$$

$$\text{and } C = 0.945 + 0.00871\ (A/0)^{4.4}$$

$$\text{Therefore, } (\text{H.T.U.}) = 0.0945 + 0.004\ (a)(f) + 0.00871\ (A/0)^{4.4}$$

where

A = feed rate of aqueous solution in cc/minutes

0 = feed rate of solvent in cc/minutes

A/0 = phase flow ratio

a = amplitude in inches

f = frequency in cycles per minute

This equation can be only used to calculate H.T.U. when the range of through-put rate was from 0 to 200 gal/ft^2 hr.

Column Hold Up

The column hold up used in the work appeared to be nearly constant. The effect on the value of H.T.U. has been neglected.

CONCLUSIONS AND RECOMMENDATIONS

From the equilibrium data and mathematical equation of correlation it has been concluded that:

The optimum extraction with regard to H.T.U. for this system was accomplished by using low arithematic product of frequency and amplitude which range was approximately 2 to 3; lower values of (a)(f) gave higher values of H.T.U.

For example:

$$\text{\% benzoic acid recovered when (a)(f) equals 3.5 (run no. 1)} = \frac{0.0092145 - 0.001157}{0.0092145} = 87.5\%$$

$$\text{\% benzoic acid recovered at (a)(f) = 9 (run no. 3)} = \frac{0.0085 - 0.00183}{0.0085} = \frac{0.00667}{0.0085} = 78.5\%$$

The percent of benzoic acid recovered would be dropped to 10% when (a)(f) product increased from value 3.5 to 9 with constant phase flow ratio.

The lower phase flow ratio (A/O), the better the extraction, if (a)(f) value was held constant. For example:

$$\text{\% of recovery of benzoic acid at}\left[(a)(f) = 3.5 \text{ with } A/O = 1.04\right] = 87.5\%$$

$$\text{\% of recovery of benzoic acid } \left[(a)(f) = 3.5 \text{ with } A/O = 2\right] \text{ (Run no. 26)} = \frac{0.00947 - 0.00279}{0.00947} = 70.6\%$$

The percent of benzoic acid recovered would be dropped to 17.5% by increasing ratio from the value 1.04 to 2.

There appeared to be but very little effect on the value of H.T.U. if the change of through-put rate was from 20 to 200 gal/ft^2 hr.

As far as operating temperature was concerned (over 62° to 91°F), the lower the temperature the larger the value of percent acid recovered. At low temperatures the equilibrium distribution coefficient was larger than at higher temperatures. There would also be but little effect on the number of transfer H.T.U. units. At lower temperatures the distance between operating line would also increase. The first and third factors would increase the H.T.N., but the second factor would decrease the value of H.T.N. and would approximately cancel each other.

The low value of H.T.U. (and of the height of an equilibrium stage) indicated that the pulse column may be made shorter than the equivalent

stationary one. This feature is particularly valuable in cases where the process demands a large number of equilibrium stages, since under these conditions a conventional column may be inordinately high.

The pulse column is more flexible than its stationary counterpart. For this reason it will be better able to handle fluctuations and changes in flow with minimum change in product quality or recovery.

The main operating cost for this equipment is power required to pump the liquid as feed into column. The additional cost is the small amount of power required for timer and cost for compressed air. It will be much cheaper than other methods of agitating extraction column.

It might be recommended for future studies that relationship between H.T.U. and higher values of through-put rate should be investigated for direct industrial application. The column should be operated without pulsing, i.e., as a single sieve plate unit, and the resulting H.T.U. values compared with those obtained when it is operated as a pulse column. Comparison can be made between pulse columns and other agitating type columns such as the York-Scheibel column and so on. The column itself might be changed with different column diameter, different plate spacing and differences in hole size of plate and percent of free area. Also, this change might allow a study for the relationship between H.T.U., diameter of column and plate geometry. Studies might also be made to change better pumping which would be helpful in getting better results. These are important factors of continuous flow and high purification in industrial application of this extraction study.

Table 6. Operating data.

Run No.	Operating Room Temp. (°F)	Air Pressure Used	Frequency f, (cycles per min.)	Amplitude a, 1/16 inch	Product (a)(f)	Flow Rate of Water A, (cc/min)	Flow Rate of Solvent 0, (cc/min)	Through-put Rate, A+0 (cc/min)	A+0, (Gal. per ft^2 hr)	Flow Ratio A/0	Concentration of Feed (x_2)	Concentration of Raffinate (y_2)	Concentration of Extract (x_1)	Concentration of Solvent (y_1)	Type of Operation	H.T.N.	H.T.U. (ft.)	^{4} H.T.U.
1	89.0	5	6.5	9	3.65	29	28	57	41.6	1.04	0.0092	0.0092	0.0011	0.0014	M.S.	5.70	0.702	0.920
2	81.5	5	8.0	9	4.50	29	28	57	41.6	1.04	0.0085	0.0074	0.0018	0.0045	M.S.	4.60	0.869	0.970
3	80.5	5	16.0	9	9.00	29	28	57	41.6	1.04	0.0085	0.0074	0.0013	0.0005	M.S.	4.30	0.930	0.984
4	71.0	8	6.5	11	4.47	80	50	130	95.0	1.60	0.0086	0.0159	0.0014	0.0024	E.	4.00	1.000	1.000
5	68.0	9	10.0	15	9.40	80	50	130	95.0	1.60	0.0085	0.0132	0.0019	0.0026	E.	3.30	1.210	1.050
6	72.5	8	16.0	15	15.00	80	50	130	95.0	1.60	0.0081	0.0120	0.0018	0.0020	E.	3.00	1.330	1.080
7	67.0	10	6.5	21	6.10	80	60	140	102.0	1.33	0.0095	0.0139	0.0016	0.0024	E.	4.20	0.952	0.992
8	70.5	10	9.6	17	11.20	80	60	140	102.0	1.33	0.0084	0.0101	0.0015	0.0008	E.	3.60	1.111	1.028
9	68.0	10	19.0	19	15.40	80	60	140	102.0	1.33	0.0077	0.0094	0.0018	0.0013	E.	3.20	1.220	1.055
10	67.0	4	6.5	7	2.85	80	20	100	73.0	4.00	0.0084	0.0215	0.0026	0.0001	M.S.	3.70	1.080	1.022
11	71.0	4	9.0	6	3.36	80	50	130	95.0	1.66	0.0092	0.0153	0.0013	0.0022	M.S.	4.20	0.952	0.992
12	76.0	4	17.0	7	7.45	80	40	120	87.5	2.00	0.0093	0.0166	0.0016	0.0013	M.S.	2.70	1.480	1.105
13	70.0	4	6.5	7	2.85	80	40	120	87.5	2.00	0.0097	0.0178	0.0023	0.0021	M.S.	2.50	1.600	1.130
14	74.0	6	9.2	11	6.32	80	40	120	87.5	2.00	0.0097	0.0166	0.0025	0.0030	E.	2.30	1.740	1.150
15	64.0	6	18.0	11	12.40	80	65	145	106.0	1.51	0.0095	0.0173	0.0015	0.0047	E.	3.50	1.143	1.039
16	69.0	6	12.0	11	8.28	80	60	146	106.0	1.66	0.0096	0.0213	0.0018	0.0047	E.	3.30	1.210	1.050
17	74.0	7	6.5	8	3.25	80	40	120	87.5	1.80	0.0143	0.0311	0.0023	0.0041	M.S.	4.00	1.000	1.000
18	76.0	5	9.0	9	5.07	80	40	120	87.5	2.00	0.0143	0.0261	0.0319	0.0038	M.S.	3.10	1.290	1.067
19	75.0	6	9.0	11	6.02	80	40	120	87.5	2.00	0.0138	0.0184	0.0033	0.0036	M.S.	2.30	1.740	1.155
20	68.0	5	6.5	7	2.85	80	40	120	87.5	2.00	0.0098	0.0193	0.0020	0.0037	M.S.	3.90	1.035	1.005
21	73.0	5	6.5	9	3.65	80	40	120	87.5	2.00	0.0092	0.0169	0.0311	0.0048	M.S.	2.17	1.840	1.160
22	68.5	7	6.5	14	5.70	80	40	120	87.5	2.00	0.0092	0.0153	0.0032	0.0035	E.	2.30	1.740	1.155
23	73.0	9	6.5	34	7.30	80	40	120	87.5	2.00	0.0090	0.0150	0.0031	0.0031	E.	2.20	1.820	1.165
24	76.0	10	6.5	21	8.52	80	40	120	87.5	2.00	0.0090	0.0173	0.0033	0.0058	E.	2.20	1.820	1.165
25	75.0	4	8.5	7	3.70	80	40	120	87.5	2.00	0.0095	0.0165	0.0028	0.0035	M.S.	2.70	1.480	1.111
26	68.0	5	8.5	9	4.80	80	40	120	87.5	2.00	0.0095	0.0152	0.0031	0.0054	M.S.	2.30	1.720	1.150
27	73.0	7	8.5	15	8.00	80	40	120	87.5	2.00	0.0092	0.0174	0.0032	0.0054	E.	2.40	1.670	1.135
28	75.0	6.5	8.5	14	7.45	80	40	120	87.5	2.00	0.0089	0.0114	0.0023	0.0023	E.	2.40	1.670	1.135
29	78.5	8	8.5	18	9.66	80	40	120	87.5	2.00	0.0090	0.0098	0.0061	0.0033	Flooding	----	-----	-----
30	80.0	8	8.5	22	11.70	80	40	120	87.5	2.00	0.0090	0.0101	0.0065	0.0036	Flooding	----	----	----
31	75.5	6	12.25	13	9.95	80	45	125	91.2	1.70	0.0087	0.0168	0.0016	0.0030	M.S.	3.80	1.050	1.010
32	75.0	7	12.25	15	11.50	80	43	123	89.6	1.76	0.0087	0.0154	0.0022	0.0030	M.S.	3.20	1.250	1.065
33	71.0	8	12.25	16	12.50	80	40	120	87.5	2.00	0.0089	0.0166	0.0031	0.0045	E.	2.20	1.820	1.165
34	74.0	9	12.25	20	15.30	80	40	120	87.5	2.00	0.0086	0.0153	0.0034	0.0042	E.	2.10	1.900	1.190
35	75.0	3	16.	7	7.00	80	40	125	91.2	2.00	0.0089	0.0180	0.0030	0.0037	M.S.	2.30	1.720	1.150
36	75.0	4	16	9	9.0	80	45	125	91.2	1.75	0.0097	0.0199	0.0025	0.0037	M.S.	3.40	1.18	1.045
37	74.5	5	16	11	11.0	80	40	120	87.5	2.00	0.0094	0.0174	0.0033	0.0051	E.	2.30	1.74	1.160
38	74.5	6	16	13	13.0	83	43	126	92.0	2.00	0.0094	0.0149	0.0037	0.0058	E.	2.00	2.00	1.190
39	74.5	7	16	15	15.0	80	40	120	87.5	2.00	0.0094	0.0190	0.0037	0.0068	E.	2.70	1.48	1.118
40	76.5	8	16	17	17.0	80	40	---	----	----	----	----	----	----	Flooding	----	----	----
41	67.0	9	16	20	20.0	80	40	---	----	----	----	----	----	----	Flooding	----	----	----
42	82.0	7	16	16	16.0	140	57	197	143.5	2.46	0.0082	0.0121	0.0048	0.0035	E.	1.10	3.64	1.387
43	80.0	4	20	9	11.2	86	43	129	94.1	2.00	0.0088	0.0127	0.0042	0.0034	M.S.	1.60	2.50	1.260
44	82.0	7	20	18	22.5	90	41	131	95.6	2.20	0.0090	0.0153	0.0039	0.0021	E.	1.80	2.22	1.240
45	83.0	9	20	22	27.5	96	48	144	105.0	2.00	----	----	----	----	Flooding	2.00	2.00	1.190
46	84.0	3	20	6	7.5	90	45	135	98.5	2.00	0.0089	0.0146	0.0041	0.0046	E.	2.10	1.90	1.170
47	83.0	4	20	10	12.5	115	71	186	135.6	1.63	0.0099	0.0131	0.0190	0.0020	E.	3.00	1.33	1.060
48	84.0	6.5	20	16	20.0	115	75	190	138.6	1.50	0.0098	0.0167	0.0252	0.0029	E.	4.00	1.00	1.000
49	82.0	9	20	21	28.8	108	86	194	141.5	1.25	0.0097	0.0166	0.0028	0.0041	E.	4.30	0.93	0.984
50	81.0	2.5	20	5	6.3	118	77	195	142.2	1.50	0.0097	0.0152	0.0027	0.0024	M.S.	4.00	1.00	1.000
51	80.0	2.5	16	6	6.0	116	65	181	132.0	2.00	0.0099	0.0099	0.0032	0.0053	M.S.	3.20	1.25	1.062
52	82.0	4	16	10	10.0	121	69	190	138.0	1.75	0.0097	0.0144	0.0037	0.0418	M.S.	2.90	1.38	1.080
53	82.0	5.2	16	13	13.0	118	70	188	137.0	1.68	0.0095	0.0171	0.0039	0.0071	E.	3.20	1.60	1.062
54	81.0	7	16	16	16.0	126	81	207	151.0	1.47	0.0100	0.0160	0.0027	0.0044	E.	3.50	1.14	1.039
55	78.0	2.5	12.5	6	4.7	117	71	188	137.2	1.65	0.0099	0.0166	0.0027	0.0046	M.S.	4.00	1.00	1.000
56	82.0	4	12.5	10	7.8	123	91	214	156.0	1.35	0.0098	0.0132	0.0026	0.0034	E.	4.00	1.00	1.000
57	80.0	2.5	12.5	6	4.7	125	83	208	152.0	1.50	0.0097	0.0159	0.0032	0.0057	M.S.	3.90	1.02	1.008
58	78.0	4	12.5	10	7.8	116	74	190	138.5	1.57	0.0096	0.0150	0.0028	0.0043	E.	3.60	1.11	1.030
59	78.0	2.5	9	6	3.4	116	83	199	145.0	1.40	0.0097	0.0093	0.0030	0.0061	M.S.	3.80	1.05	1.010
60	79.0	4	9	10	5.6	121	73	194	141.5	1.63	0.0097	0.0163	0.0027	0.0046	M.S.	4.00	1.00	1.000
61	81.0	5.5	9	14	7.9	119	83	201	147.5	1.47	0.0096	0.0016	0.0032	0.0063	M.S.	4.00	1.00	1.000
62	82.0	2.5	6.5	6	2.4	124	88	212	155.5	1.47	0.0096	0.0160	0.0035	0.0068	M.S.	3.70	1.08	1.020
63	86.0	4	6.5	12	4.9	118	83	201	146.7	1.47	0.0096	0.0160	0.0035	0.0070	M.S.	3.80	1.05	1.010
64	86.0	5.5	6.5	16	6.5	122	86	208	152.0	1.47	0.0097	0.0180	0.0033	0.0071	M.S.	3.90	1.02	1.005
65	86.0	4	6.5	12	4.9	127	128	255	186.0	1.00	0.0096	0.0104	0.0024	0.0031	M.S.	4.50	0.89	0.970
66	93.0	5	11.5	16	11.5	124	60	184	134.0	2.07	0.0091	0.0171	0.0036	0.0037	E.	3.40	1.18	1.042
67	95.0	4	11.5	10	7.2	138	97	235	117.5	1.42	0.0088	0.0042	0.0113	0.0047	M.S.	2.50	1.60	1.100
68	95.0	2.5	11.5	6	4.3	124	83	207	151.0	1.50	0.0092	0.0042	0.0159	0.0083	M.S.	3.90	1.02	1.005
69	88.0	4	11.5	10	7.2	125	113	238	178.0	1.20	0.0099	0.0030	0.0159	0.0077	M.S.	3.70	1.08	1.010
70	89.0	4	20	10	12.5	125	118	243	177.0	1.15	0.0098	0.0024	0.0119	0.0034	E.	4.50	0.89	0.975
71	85.0	2.5	20	6	7.5	125	122	247	180.0	1.02	0.0101	0.0020	0.0019	0.0040	M.S.	4.60	0.87	0.970

ACKNOWLEDGMENTS

The author wishes to thank Kansas State College and the Department of Chemical Engineering for their financial assistance and the use of their laboratory equipment.

Acknowledgments are also extended to:

Dr. Henry T. Ward for his consultation and guidance during the writing of the thesis.

Dr. Shu-lung Wang for his time and assistance in the assembly of the column and the foundation of the field of study.

BIBLIOGRAPHY

1. Brown, G. E. and C. W. Kuhlman
   A process for recovery of uranium from pitchblende disgestion residues. Chem. Eng. Sym. No. 13, 50, 5, 1954.

2. Boegli, Jacques S.
   The separation of cadium and copper salt by liquid-liquid extraction. M. S. thesis, 1953, Kansas State College.

3. Burns, W. A., A. C. Groot and C. M. Slansky
   Atomic Energy Commission reports, HW-14728, Oct. 1949.

4. Cohen, R. M. and G. H. Beyer
   Performance of a pulse extraction column. Chem. Eng. Prog., 49, 279, 1953.

5. Fales, H. and W. Kenny
   Inorganic quantitative analysis. Chap. VIII, pp. 205-222. New York: Appleton Century Co., 1939.

6. Feick, George and H. F. Anderson
   Performance of a packed liquid-liquid extraction column under controlled agitation. Ind. Chem. 44, 404, 1952.

7. Griffith, W. L., G. R. Jasny, and H. T. Tupper
   Atomic Energy Commission declassified report. AECD-3440, 1952.

8. Harman, Robert E.
   A continuous liquid-liquid extraction for organic solvents denser than water. J. Chem. Educ. 30, 261, 1953.

9. Jan, Rydberg
   The extraction of metal complex between thorium and acctylaceton. Arkiv Kemi 5. 413-23, 1953.

10. Li, W. H. and W. M. Newton
    Liquid-liquid extraction in a pulsed perforated plate column. A.I.Ch.E. Journ. 3, 56, 1953.

11. Liquid-liquid extractor. Chem. Eng. 61, 286, Jan. 1954.

12. Mickley, H. S. and T. K. Sherwood
    Mathematical analysis in chemical engineering. Chap. I, pp. 1-42. New York: McGraw-Hill Book Company, Inc.

13. Pigford, R. Lt., et al.
    Absorption and extraction, sec. ed. Chem. Eng. Series, Chap. V, pp. 115-162, 1952.

14. Sege, G. and F. W. Cohle
    Pulse column variables. Chem. Eng. Sym. No. 13, 50, 179, 1954.

15. Solvent extraction takes hold. Chem. Eng. News, 35, 88, March 1957.

16. Thornton, T. D.
Recent development in pulsed column techniques. Chem. Eng. Sym. No. 13, 50, 39, 1954.

17. Van Dijck, W. J. D.
U. S. Patent 2011, 186, Aug. 1935.

18. Von Berg, R. L. and H. F. Wiegandt
Surge in liquid cyclones. Chem. Week, 69, 31, Dec. 15, 1951.

19. Warner, R. K.
Solvent extraction of uranium, thorium and copper nitrates. Australian J. Appl. Sci. 4, 427-43, 1953.

20. Woodfield, F. W. and G. Sege
A lower plsyr redistribution for large diameter pulse columns. Chem. Eng. Sym., No. 13, 50, 14, 1954.

APPENDIX

Overall material balance on basis of gram-moles/liter for run (1)

top layer:

Concentration of benzoic acid in raffinate (y_2)= 0.0096033 gram mole/ liter

Concentration of benzoic acid in solvent (y_1) = 0.00136 gram mole/ liter

bottom layer:

Concentration of benzoic acid in feed (x_2) = 0.0092145 gram mole/liter

Concentration of benzoic acid in extract (x_1) = 0.001157

By the material balance:

$$(y_2 - y_1) = \frac{A}{0}(x_2 - x_1)$$

$$\frac{A}{0} = \frac{(y_2 - y_1)}{(x_2 - x_1)}$$

$$= \frac{0.0082433}{0.0080576}$$

$$= 1.02$$

This value is very close to the value 1.04 measured as flow ratio.

$$\% \text{ of error} = \frac{1.04 - 1.02}{1.04}$$

$$= \frac{0.02}{1.04}$$

$$= 0.00182$$

$$= 0.1827\%$$

THE EXTRACTION OF BENZOIC ACID IN A PULSE COLUMN

by

CHANG-LO WU

B.S. The Ordnance Engineering College, 1953
Taipei, Formosa

AN ABSTRACT OF A THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Chemical Engineering

KANSAS STATE COLLEGE
OF AGRICULTURE AND APPLIED SCIENCE

1958

Liquid extraction of benzoic acid-water solution by means of toluene were studied by varying phase flow ratio of both feed and solvent in 2-inch diameter pulse column to which different frequency and amplitude were applied. Studies on extraction process of both continuous feed and solvent by making toluene solvent as dispersion phase through aqueous continuous phase. With fixed flow ratio of both solvents the optimum operation was obtained by using lower (a)(f) product. If (a)(f) value were held to be constant, the lower the phase flow ratio, the better the extraction. Data on the through-put rate are included. The results are correlated by the following equations:

$$H.T.U. = (0.004\ af + C)^4$$

and

$$C = 0.945 + 0.00871\ (A/0)^{4.4}$$